6. APPLICATION OF INNOVATIVE MATERIALS

A. Advanced Materials for Friction Brakes

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objectives

- Identify, test, and analyze the friction and wear characteristics of advanced materials and surface treatments that enable weight reduction in truck brake components while equaling or bettering their performance. Materials of interest include intermetallic alloys, ceramic composites, titanium (Ti) alloys, and novel surface treatments.

Approach

- Develop and build a subscale brake material testing apparatus that will enable friction studies of advanced materials at speeds and pressures similar to those experienced by full-sized brakes.
- Investigate the nature of changes to the surfaces of materials that occur as a result of frictional contact under high-energy-input conditions. Relate these to material properties.
- Develop an improved understanding of the role of friction films in vehicle braking and apply that knowledge to the exploration of promising new truck brake materials.
- Examine the characteristics of brake material wear particles and their role in total vehicle emissions.

Accomplishments

- Designed and built a subscale brake testing apparatus instrumented to record applied force, friction force, surface temperature, and vibration.
- Selected and tested a variety of materials with the potential to serve as lightweight brake components.
- Investigated the effects of wet and dry braking conditions on frictional behavior.
- Used infrared imaging to study frictional heating of both cast iron and ceramic brake materials.
- Evaluated the friction and wear compatibility of metals, polymers, and ceramics with Ti alloys.

- Evaluated the effects of surface roughness on the friction of commercially-coated Ti alloys.
- Characterized wear particles that are generated in tests of candidate brake materials.

Future Direction

- Evaluate currently-available commercial coatings and explore new coating technologies for use on Ti alloys for use as lightweight brake rotor materials.
- Identify appropriate materials that are frictionally compatible with Ti or coated Ti alloys.

Introduction

New, more aerodynamic designs for energy-efficient heavy trucks can improve their fuel economy. Advanced tires with lower rolling resistance can also improve energy efficiency. However, as these measures decrease the effective drag on trucks, the demands on their braking systems must increase. Braking system technology involves design, instrumentation and control, and materials aspects. This project specifically addresses the latter. Brake materials must exhibit a balance of properties, including frictional stability over a wide temperature range, good thermal transport, dimensional stability, corrosion resistance to road de-icers, and wear resistance. From a practical standpoint, they must also be cost-competitive. Opportunities exist to employ advanced materials to create lighter-weight braking systems that will enable new technologies to raise the fuel efficiency of a vehicle without compromising its safety and reliability.

This project is aimed at evaluating advanced structural materials and surface treatments that show potential as truck brake friction materials. Testing such new materials is made more cost-effective by using small specimens to screen the most promising candidates. To this end, a subscale brake tester (SSBT) was designed and built. It was instrumented to measure normal force, friction force, surface temperature, and vibrations during braking. An attachable water spray system enables study of the effects of wet and dry braking. The SSBT has been a workhorse in recent studies involving a variety of both traditional and nontraditional brake materials. Test results are supplemented by optical microscopy, electron microscopy, and transmission electron microscopy to study friction-induced film formation.

Candidate Materials

Studies of the structure, composition, and physical properties of candidate materials eliminated low-melting-point, brittle, corrosion-prone, and environmentally unacceptable materials. A list of evaluated materials, with brief comments about them, is presented in Table 1. Typical SSBT specimens of these materials are shown in Figure 1. Dark annular rings on these 5-in.-diameter disc specimens show the thin films that are produced by sliding contact. Films like these also develop on the opposing brake lining materials.[1]

Of the materials in Table 1, it was decided to focus on Ti alloys for several reasons: new, cost-effective methods for processing Ti are being developed; resistance to corrosion by road de-icing compounds is very good; and the high-temperature strength of Ti alloys is better than that of aluminum matrix composites. In addition, one manufacturer of Ti alloy discs for racing vehicles (Red Devil Brakes, Mt. Pleasant, PA) has developed a promising coating process for Ti and provided several brake pad compositions for evaluation.

Since tribology data for Ti are relatively rare, preliminary friction and wear tests were

Table 1. Issues associated with certain candidate materials for lightweight truck brakes

Material	Issues
Traditional gray cast iron and commercial brake pads	Served as a reference with which to compare data for other materials
Aluminum matrix-based composite materials containing silicon carbide hard particles	Lightweight, but low melting point creates concerns for use in heavy trucks with hot brakes; attacked by road de-icers
Intermetallic alloy based on Fe_3Al	Modest weight saving; not commercially available at low cost; wears by cutting chip formation (metallic flakes)
Ceramic composite of carbon and silicon carbide	Expensive; good frictional performance at high temperatures; fibrous wear particles produced; machining and handling present some problems
Titanium alloys	Lightweight and stiff; very good high-temperature strength and corrosion resistance to salts; considerable data from aerospace R&D; poor wear characteristics and low thermal conductivity

Figure 1. Candidate brake disc materials after friction testing on the SSBT: commercially-coated Ti (upper left), experimental ceramic composite (upper right), uncoated Ti alloy (bottom).

performed on two Ti alloys provided by TIMET Corporation (Ti-6Al-4V and Ti-6Al-2Sn-4Zr-2Mo). The first is a workhorse aerospace alloy, and the second was designed for higher-temperature applications. A fixed sphere (pin) sliding on a rotating, flat disk was used as the test geometry for initial screening. Table 2 summarizes the results of these experiments on the Ti-6Al-4V alloy. Similar data were obtained with the other alloy. In examining the data, it should be noted that most braking systems operate within friction coefficients of between about 0.35 and 0.55.

Table 2. Average sliding friction coefficient (μ) of pins of various materials sliding on Ti-6Al-4V alloy disks.

Pin material	μ at 0.3 m/s	μ at 1.0 m/s
440C stainless steel	0.50	0.35
Alumina ceramic	0.47	0.36
Silicon nitride ceramic	0.49	0.44
PTFE (Teflon™)	0.28	0.29

[a] Load 10 N, sphere diameter 9.53 mm, air, room temperature.

While friction data for all materials but the PTFE seemed reasonable for brakes, wear data from these tests, reported elsewhere, were unacceptably high. Thus surface treatment or coating will be needed to make Ti a viable brake material. Larger-scale pad-on-disc tests were performed on the SSBR to compare bare Ti-6Al-4V alloy with commercially coated Ti-6Al-4V alloy cut from the same billet.

Subscale Pad-on-Disc Tests of Titanium

The SSBT was used to conduct higher-speed sliding friction tests on both coated and uncoated Ti discs. Coated discs were thermally sprayed with a proprietary ceramic particulate composite in a metallic binder (Red Devil Brakes). A semi-metallic, racing-grade pad material from the same commercial source was used. The effects of applied pressure, speed, and surface finish were investigated. As shown in Figure 2, friction of the as-coated Ti was higher and somewhat more variable, but it was more in the range for typical vehicle brakes. Optimization of the surface finish and more rigorous "burnishing" can produce a stable friction layer that reduces such frictional fluctuations. The coated brakes ran slightly hotter, but their wear was so small as to be unmeasurable in the current experiments. Additional research on Ti-based materials, exploring alternative coatings, is planned for FY 2004. Attention will be paid to the role of friction-induced surface films and the selection of a suitable pad material.

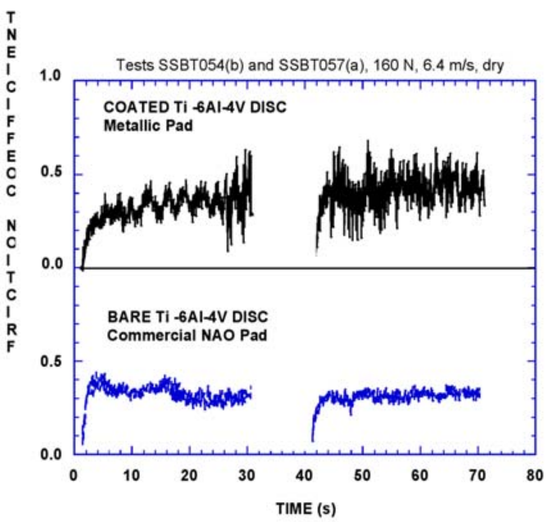

Figure 2. Friction traces for tests on bare and coated titanium for two pad applications at a sliding speed that equates to about 30 mph on a full-sized truck.

Conclusions

Studies have been conducted of a variety of candidate lightweight truck brake materials. Of these, Ti alloys offer a number of attractive characteristics, but their low thermal conductivity and wear behavior must be enhanced. The use of surface coatings or treatments is expected to enable the materials to perform quite well as brakes. Further work in FY 2004 is planned to better understand the friction and wear behavior of Ti alloys and to evaluate several alternative coating methods.

References

1. P. J. Blau. "Microstructure and Detachment Mechanism of Friction Layers on the Surface of Brake Shoes," *J. of Matls. Engr. and Performance*, *12*(1), 56–60, 2003.

B. Advanced Composite Structural Cab Components

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Contractor: Delphi Corporation
Contract No.: 4000009401

Objective

Develop an advanced composite cab structural component for a Class 8 tractor:

- Develop the design, materials, and manufacturing process for utilizing continuous-oriented, fiber-reinforced composites for affordable commercialization within 3 years of beginning the project.
- Reduce the existing mass by at least 30% from 22.8 kg to 15.96 kg.
- Meet or exceed the performance of existing materials.

Approach

- Organize a value analysis/value engineering (VAVE) workshop to generate different options for design and manufacturing.
- Develop finite element (FE) analysis for predictive engineering and develop design options.
- Perform a cure-cycle study, make necessary panels, and study Class A surfaces.
- Conduct design failure modes effects and analysis (DFMEA).
- Construct prototype parts and verify proposed design using a design validation (DV) test.
- Release final design, construct production tools, and validate the production phase.
- Begin production.

Accomplishments

- Held VAVE workshop, generating options for design and manufacturing.
- Developed FE analysis models, using proposed materials to predict part performance.
- Performed cure-cycle study, made composite panels, conducted Class A activities
  - Conducted DFMEA.
  - Constructed tools for prototype constructions.
  - Made prototype parts.

Future Direction

- Complete DV test.
- Refine FE models using new information from DV test.
- Develop final design and process.
- Release final design, construct production tools, and validate the production phase.
- Begin production.

Introduction

Weight can be reduced and fuel efficiency increased in trucks and tractors, without compromising structural integrity or utility, by incorporating innovative designs that strategically utilize modern lightweight materials.

The Advanced Composite Structural Cab project aims to reduce the mass of a Class 8 tractor cab structure while maintaining its performance by using oriented fiber technology that is typically used in the aerospace industry. The selected structure is a chopped-glass, reinforced-vinyl ester approximately 3 mm thick with nonoriented 55% chopped-glass mat preform and local steel backing. The trim panel is 27% glass-filled sheet molding compound (SMC) in a polyester matrix.

Value Analysis/Value Engineering

The team conducted a VAVE workshop with the original equipment manufacturers (OEMs) in September 2000. Baseline design options and ideas were generated at this workshop. These ideas were used to start the three-part design. When the team chose to consider a two-part design, a few more mini-VAVE workshops were conducted. Technical experts generated lists of new ideas and technologies at these workshops; some of those concepts were used to define design options.

Finite Element (FE) Analysis Models and Design Options

The OEMs provided IGES files and drawings for the current production part, along with various fasteners used in the part. Based on performance requirements and the FE results, key load case performance criteria such as sag and twist were identified. Twelve load cases were considered for the design.

Initially, a three-part design was considered. Several design options were created and optimized for weight and performance using this model. However, the design did not meet the weight and cost targets. A two-part design option was presented to the project team during the February 13, 2003, quarterly meeting. The project team agreed to pursue a two-part design.

Several options for two-part design were created; Table 1 shows the weight target comparison. The project team decided to use the TA3-5 option to start prototype activities even though it did not quite provide a 30% weight reduction.

The design team is still working on more options, including options to reduce the flange area to 3.0 mm, use foam for reinforcement shapes, and create metal reinforcements that will help reduce cost and weight.

Cure-Cycle Study

In order to determine the proper tooling and cure cycle, a potential resin supplier conducted a cure-cycle study. The goal is to make a resin recipe that will meet a 22-min full-cure cycle at 120°F. The 120°F cure cycle

Table 1. Design matrix

Design option	TA-1-2	TA-3-1	TA3-1-rev1	TA-3-2	TA3-3	TA3-5
Total Weight Kgs	**19.71**	**16.46**	**17.09**	**17.36**	**16.82**	**17.03**
Weight Savings	9%	24%	21%	20%	23%	25%
Performance	**Comparable to baseline**	**Improved performance**	**Improved performance**	**Improved performance**	**Improved performance**	**Improved performance**
Trim Panel mass Adhesive	Thinner 0.294kg Plexus	Existing SMC 0.7kg Plexus	Existing SMC 0.7kg Plexus	Existing SMC 0.7kg Plexus	Existing SMC 0.7kg Plexus	Existing SMC 0.7kg Plexus
Fabric	Quad	Tri#	Tri #	Tri #	Tri **	Tri **
Flange area thickness	6.00 mm *	6.00 mm	6.00 mm	6.00 mm	6.00 mm	6.00 mm
Foam Core density	Single pc 4lb/ft3	Multi pc 4lb/ft3	Multi pc 4lb/ft3	Multi pc 4lb/ft3	Multi pc 4lb/ft3	Multi pc 4lb/ft3
Hinge Reinforcement	Existing - 1 pc 3.18 mm	***HR3-1*** design in 5 pcs	***HR3-1*** design in 5 pcs	***HR3-1*** design in 5 pcs	***HR3-1*** design in 5 pcs	***HR3-1*** design in 5 pcs
Ribs	***Unribbed***	***0.81-1.55 mm***	***1.5mm***	***1.23-1.66 mm***	***0.50- 2.43 mm***	***0.70- 2.17mm***
Attachment points with Fasteners	**Existing**	**Existing**	**Existing**	**Existing**	**Existing**	**4 pcs Existing 2pcs thin, Latch area extened**
Risk	Low	Low	Low	Low	Low	Low

6.00mm * =3.0.mm composite material plus 3.00 mm of resin only to make it 6.00 mm
 # = Specialty fabric- special order required
**** = Off-the shelf fabric**

will present less degradation of the composite tool yet meet the intended production volume. Cure-cycle curves will be prepared at room temperature, 120ºF, and 140ºF. The expected completion of this study is December 2003.

Composite Panels

The manufacturing constraint of making oriented 1.5-mm thin panels was addressed by making panels with a 30% fiber volume. The significance of this is that the fiber volume used is closer to that of traditional molding practices using nonoriented fiber, yet it allows panel thickness reduction from the traditional 3.0 mm to 1.5 mm. Three sets of panels using the resin and process intended for production were made, and their physical properties are being evaluated.

Class A Surface Activities

The development of a Class A surface for structural components using three-dimensional woven fabric was conducted. Three trials were conducted using the resin transfer mold (RTM) process with three different sources and three different types of tools (aluminum, composite, and multiple insert tooling). Each of the three used a low-profile polyester resin system known for its Class A quality. Each achieved the best surface quality so far using a similar laminate lay-up and an in-mold coating. The initial studies showed that a greatly improved structure using the same amount of material can be achieved.

The Class A activities were suspended because more time and resources are needed. A current production part or equivalent will be utilized for this project.

Design and Process Failure Modes Effects and Analysis

A system-based DFMEA was initiated at the Delphi facility. The team conducted another DFMEA with OEM engineers. The process is being continued with the ranking of all items. A follow-up DFMEA will be conducted with the OEM engineers after the DV tests are completed. Potential suppliers will be responsible for performing process failure modes effects and analysis.

Prototype Activities

Two composite tools using the TA3-5 design concept were made in Delphi's Salt Lake City labs in June 2003. The first tool was made for composite parts, and a second tool was made for foam.

As no computer-assisted design data were available at that time, the tools were made from the current production part using splash. Unfortunately, that technique could not accommodate the shrinkage factor for the foam tool. Another difficulty was that the tool was made using composite material as opposed to the normal practice of using aluminum tools, because of time and cost factors. An external heating element was required to make good-quality parts. However, the tool design did not permit us to add the plumbing needed. External heating blankets were utilized to overcome this difficulty. First, the foam supplier made some foam sets without hardware to set up the process parameter. Then more sets of foam sets were delivered with hardware molded in.

Starting July 14, 2003, the team started making prototype parts. The first part was infused in 1.5 min. The part was about 2.0 kg heavier than design because of the foam's inconsistency. The second part was not filled completely. After the unsuccessful third trial, the team members met at the supplier's location on July 30, 2003, to resolved this problem, and they decided to use a low-pressure vacuum infusion method. Prototype No. 5 was made using foam without any hardware. It was completely filled and looked good overall. Most of the regions had composite thickness as intended. However, there were some areas where composite panels were thicker than intended. This was the result of foam being under-sized. The foam intended for production will be made using metal tools, and that should solve this problem.

The team decided to drill holes in the composite tools to locate all metal attachments. During the first week of August 2003, the composite tool was sent for a coordinate measurement machine (CMM) check. Based on the CMM reading, holes were drilled, and the tool was prepared to make prototype parts with hardware.

Prototype part 6 leaked in one area and was not filled completely. Prototype 7 did not fill in one small place. Prototype 8 filled to almost 95% and looked good. The team decided to send this part for a CMM check. The CMM check on the part revealed that three areas were slightly off because the tool was warped. A fixture was made to correct this problem. Prototypes 9 and 10 did not fill as desired, especially in one particular area on the top of the part. The team believes that this problem can be corrected by adding a flexible seal in the area where the part does not fill. The team also decided to bond metal strips on the backside of the tool to provide uniform pressure.

Plans for Fiscal Year 2004

The team will complete the DV tests, complete the final design, and start working on production tools. The production tools are expected to be completed around March 2004. Production validation tests are expected to be completed by June 2004. Start of production in a limited quantity is expected by July 2004. By the end of September 2004, full-scale production is scheduled.

C. Advanced Composite Structural Chassis Components

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Contractor: Delphi Corporation
Contract No.: 4000009401

Objectives

- Develop an economical, long-fiber-reinforced manufacturing procedure utilizing continuous and/or oriented chopped fibers for structural chassis components for Class 7 and 8 trucks.
- Reduce mass of these components by 60%.
- Commercialize and produce these components, reducing vehicle mass by about 50 kg/vehicle and significantly increasing North American carbon fiber demand annually, within 5 years of beginning the project.

Approach

- Conduct value analysis/value engineering workshop(s) to conduct function analysis and brainstorm solutions using composites for each component function.
- Develop finite element analysis (FEA) models at both the component and system level. Conduct structural optimization (topology and shape/sizing) on components for “material efficient” designs.
- Build and test prototypes.
- Secure production orders for the components developed within the scope of the project.

Accomplishments

- Received customer production part approval of two different models of lateral links. Completed first order of more than 1000 lateral links. Submitted samples and obtained initial order from second commercial vehicle customer. Composite lateral links are 66% lighter than current steel, resulting in almost 5 kg/system mass savings.
- Completed design of proof-of-concept composite-reinforced, thin-wall steel tube main support.

- Completed fabrication and assembly of all metal components of main support. Completed molding of first composite-reinforced support.
  — Tier 1 partner used composites research and design concepts to develop and commercialize an aluminum z-beam, which resulted in over 27 kg mass savings per system. Combined with the mass savings of the lateral link, this project will have realized 32 kg of the 50 kg target for system mass reduction by the end of FY 2003.

Future Direction

- Complete building and testing the proof-of-concept reinforced main support. This product has the potential for over 20 kg additional mass savings.
- Complete the cost model for reinforced main supports.
- Select the appropriate support for commercialization.
- Investigate lower cost lateral link designs and processes.

Introduction

In response to a request for proposals from Oak Ridge National Laboratory (ORNL) in February 2001, a submission from Delphi Corporation led to the award of a subcontract for the development of advanced composite structural chassis components with the objectives listed above.

Sponsored by the DOE, the subcontract is scheduled to run for three years with an estimated cost of $2.5M. This project is a 50/50 cost share between ORNL and industry. In this project, Delphi Corporation, the world's largest automotive Tier 1 supplier, partnered with an industry-leading Tier 1 supplier to the truck and trailer industry and focused on three components in a chassis/suspension system: lateral links, main supports and z-beams.

Lateral Link Status

A key project milestone (limited-volume commercialization) for the lateral link was realized at the beginning of FY 2003 with the acceptance by Delphi of the first 1000-piece order from its Tier 1 partner. Two different models of the link (different lengths for different applications) have been approved through the production part approval process (PPAP), and several hundred are in use in the field.

Glass and standard modulus carbon-fiber-reinforced prepreg are the materials utilized in the manufacture of the links. The bulk of the material is carbon-fiber reinforced to obtain the buckling stiffness required. Tubes are mandrel roll-wrapped, cured, and threaded. Metal inserts are then bonded at each end. The glass prepreg is employed for the threading operation and protective outer layer.

Current composite links offer a 67% mass reduction and outperform the mainstream steel in buckling load capacity and three-point bending, and they also have a significantly higher natural frequency. Examples of both link assemblies are shown in Figure 1.

Although the lateral link can be deemed a success both technically and commercially, costs are still somewhat prohibitive for applications other than lower-volume niche markets. In 2003, efforts were made to develop lower-cost designs and processes to broaden the market potential and increase the impact on fleet fuel efficiency. Two options that were built as prototypes were hybrids (thin-walled steel overwrapped with composite) and pultrusion. Cost models for the hybrid design were not favorable, and costs for pultruded designs are still being analyzed. Pultruded samples are currently in

Figure 1. Typical steel (top) and composite lateral link assemblies.

design validation testing, with results expected by December 2003.

Because the properties of the composite tube are significantly superior to those of the steel, a lower-cost roll-wrapped option is also being considered. In this option, the number of carbon fiber unidirectional layers will be reduced, and the glass fabric on the inner diameter will be placed only at the ends instead of along the entire length. It is anticipated that this will yield up to a 20% cost reduction.

Delphi has requested that additional project funds be transferred from the project to the ORNL materials laboratory for continued durability and buckling testing of these lower-cost alternative designs.

Main Support Status

A significant amount of design effort was spent on the main support during FY 2003. The main thrust of this design was toward carbon-fiber reinforcement of a relatively thin-walled steel tube. Although the design of the tube reinforcement remained stable throughout the year, much difficulty was encountered in obtaining a solution for the interface between the tube and the mounting hardware and brackets.

The initial all-composite interface design was not cost-effective. When all steel hardware was used in the design, mass targets were not met. As a compromise, a hybrid interface was designed in which thin steel brackets were welded to the tube (see Figure 2). These brackets were then reinforced with composites with the rest of the tube.

The current prototype design yields a mass savings of approximately 30% or 23 kg per system.

Six steel framework subassemblies (Figure 3) were fabricated during August and September 2003. The first composite-reinforced assembly was run during the first week of October and is shown in Figure 4 prior to any finishing work.

These prototypes are fabricated by placing dry fabrics with specific fiber orientations around the tube and brackets. The entire support is then bagged and infused with epoxy resin. For production, mold tooling would be used for application of the reinforcement material.

Testing of the reinforced main supports is scheduled for November at Delphi's Tier 1 partner. Several load cases are included in this testing, including vertical beaming, braking, and side load.

Z-Beam Status

At the beginning of FY 2003, Delphi's Tier 1 partner announced its intention to commercialize an aluminum version of the

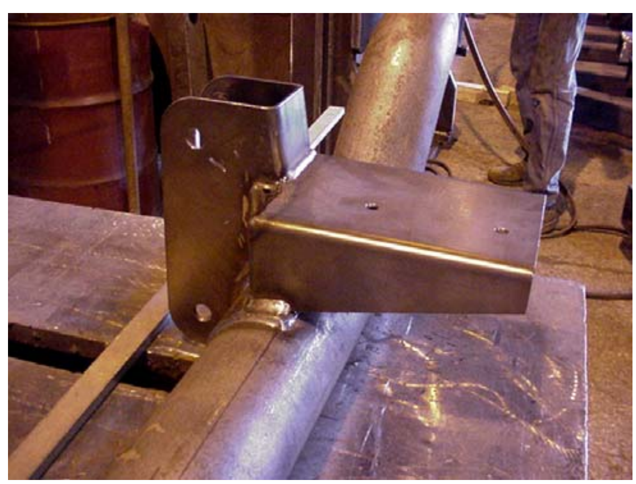

Figure 2. Steel interface-bracket assembly on subplate welded to tube.

Figure 3. Prototype steel framework ready for composite fabrication.

Figure 4. First composite-reinforced main support just out of cure oven, prior to any finishing work.

z-beam based on FEA topology optimization completed for a composite design. The cast aluminum solution reduced the mass by approximately 7 kg and was at cost parity with or below the current cost of welded steel designs. Because four z-beams are used in each system, total system mass was reduced by 28 kg.

Based on the success of the aluminum, work was suspended on composites for the first half of FY 2003. Composites were considered again for a heavy-duty application in the April thru June timeframe, but cost models were not favorable, and work was suspended again.

Plan for Fiscal Year 2004

Completion of the building and testing of the proof-of-concept prototype that used a reinforced main support will occur in FY 2004. Commercialization of this component in the chosen application is questionable because Delphi's Tier 1 partner has expressed interest in a multipiece design for its next generation of products. Unfortunately, these designs are not currently "composite-friendly." During the first quarter, alternative applications will be investigated and pursued. Delphi has already begun negotiations with a second, larger Tier 1 partner.

A minor amount of resources will be dedicated to validate lower-cost lateral links through building and testing the prototype. As higher volumes are required, funds will be required for production tooling of inserts and threading of the tube.

Because of changes in the project scope mentioned earlier, a request for contract modification will be submitted by Delphi in early November to update the budget, scope, cost-sharing schedule, milestones, deliverables, and commercialization plan. Delphi recommends that funding not used because of underspending on this project be made available for other projects.

D. Carbon Fiber Sheet Molding Compound for Class 8 Vehicle Hoods

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Contractor: Volvo Trucks North America
Contract No.: 4000010928

Objective

- Develop carbon-fiber-reinforced sheet molding compound (SMC) and processing techniques that will enable serial production of Class 8 truck hoods with structural integrity, Class A surface quality, significantly reduced mass, and competitive cost compared with existing glass fiber SMC molded components.

Approach

- Accumulate material property data to establish reliable design properties that can be utilized for engineering design analysis.
- Perform finite element analysis of a carbon fiber SMC-based, Class 8 hood design.
- Evaluate consistency and repeatability of carbon fiber SMC material properties, processing techniques, and surface quality.
- Evaluate mass savings and costs.
- Confirm predicted results by constructing prototype hoods and performing accelerated endurance tests.

Accomplishments

- Completed preliminary short beam shear tests to screen carbon fiber materials most likely to provide the desired mechanical properties.
- Completed preliminary spiral flow trials to compare molding-related parameters of materials.

- Completed preliminary evaluation of surface quality based on carbon fiber SMC molded in existing glass fiber SMC component molds.
- Initiated review of hood surface and structural component assembly and bond interfaces.

Future Direction

- Continue evaluation of carbon fiber SMC materials.
- Pursue process developments to enhance exterior panel Class A surface quality.
- Continue evaluation of assembly processes relating to adhesive materials, bond gaps, and processing.
- Follow up Class 8 truck hood costs and weights based on carbon fiber SMC material and process developments.

Introduction

The mass of light automotive and commercial heavy-duty vehicles can be reduced utilizing modern, lightweight, high-performance composite materials. The reduction in vehicle mass translates into an increase in fuel efficiency. Currently, polymeric carbon fiber composites are used in low-volume, high-performance applications such as spacecraft, aircraft, and race cars. Carbon-fiber-reinforced composites can reduce vehicle body mass by 40 to 60%. However, market conditions and technical barriers inhibit their use in high-volume automotive applications.

Class 7 and 8 trucks offer a lower production volume, lower technical barriers, and adequate financial incentives that justify a modest price premium for competent lightweight materials. The aim of this project is to accelerate the commercial implementation of high-performance, lower-cost carbon fiber SMC body components for Class 7 and 8 trucks. As utilization of carbon fiber SMC develops and the technology matures, it is foreseeable that carbon fiber SMC will migrate into the high-volume automotive market.

This project was initiated by performing a comparative finite element analysis of a hood configuration made of glass fiber SMC material that had been validated through modeling, accelerated endurance tests, and field tests. Based on expected carbon fiber physical and mechanical properties, hood structural and surface component material thicknesses were reduced through several iterations to determine the effect on hood system stress states and displacements. Modal analyses were performed to determine mode shapes, and complete vehicle models were used to obtain dynamic responses in the frequency domain. Fatigue life comparisons were made based on the complete vehicle model transient analyses.

Based on the initial investigation, it was concluded that a competent hood could be produced with a 40 to 60% reduction in hood mass if a carbon fiber SMC material could be produced that would consistently provide the physical and mechanical properties targeted.

Carbon Fiber SMC Material Search and Comparative Testing

A search was initiated to find suppliers of polymers and carbon fibers combined in a useable SMC. Materials from Zoltec, SGL, Toray, Grafil, and others were evaluated. Early on, two significant obstacles became evident: completely wetting the carbon fiber and defilamentizing the fiber bundles. Work with suppliers is ongoing to optimize chemistry and processes to consistently provide carbon fiber SMC material with the targeted material properties. Short-beam shear tests using ASTM D2344/D 2344M were used to screen material samples with various combinations of resins and additives. More than 150 samples were evaluated. Materials that performed well in the short-beam shear screening were used to make plaques to measure material properties. Good progress has been made in obtaining the targeted mechanical properties, as shown in Table 1.

Table.1. Percentage of target values achieved

Property	Tensile strength	Tensile modulus	Flexural strength	Flexural modulus
Percentage achieved	93%	91%	96%	96%
Coefficient of variation	0.05	0.09	0.09	0/07

Class A Surface Quality Development

The carbon fiber SMC material, which exhibited mechanical properties closest to the targets, was utilized in preliminary molding trials to assess the status of surface quality. Existing automotive exterior panel molds were used, and processing parameters were set similar to those for the glass fiber SMC component. The assessment of the surface quality of the initial parts appeared promising. However, a substantial effort will be required in process engineering and process optimization to achieve Class A surface quality.

Assembly Process Evaluation

Evaluations of various proposals for bond gaps, adhesives, and assembly processes were begun. At this time, no definitive direction has been reached. Work in this area will proceed concurrently with the carbon fiber SMC material and process developments.

Cost and Weight Evaluation

Initial concept work has concluded that a 40 to 60% weight reduction is within range, as plaques in the required reduced material thicknesses have been molded. Early cost estimates of the carbon fiber SMC material are encouraging. However, a great deal of work remains to ensure that material, processing, assembly, and finishing costs are competitive with complete glass fiber SMC components.

Conclusions

Good progress has been made in finding promising materials and beginning process development. The early work is very encouraging, as target material properties are being approached. Process development has begun, and assembly process investigations are ongoing. The potential cost and weight reductions appear realistic and within reach.

E. Advanced Composite Support Structures

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Contractor: Delphi, Inc.
Contract No.: 4000021806

Objective

- Lead the rapid implementation of lightweight composite materials in Class 7/Class 8 vehicles via the development of advanced composite support structures, including chassis lateral braces and primary beams.

Approach

- Research four topic areas: joining, modeling, designing and processing. Coordinate the joining task with the project “Attachment Techniques for Heavy Truck Composite Chassis Members” to leverage resources.
- Study and test various joint designs and mechanisms, including mechanical fastening and adhesives. For mechanical fastening, assess parameters such as pre-drilled versus post-drilled holes, as well as hole geometry, spacing, bolt tension, and inserts.
- For the modeling task, study predictive numerical techniques to aid in joint and overall part design. Use several computer-aided engineering vendors to determine which would best complement the fiber architecture, fatigue, and damage analyses, which are needed to accurately predict joint and part response in this application. Use commercial finite element analysis (FEA) software to optimize the part geometry, including thickness variation, fiber orientation, fiber architecture, hole location, peek performance, and the minimization of mass and cost.
- Conduct the design using the results of the joining and modeling sections to determine a process to fabricate prototype parts for testing.

Accomplishments

- Successfully completed the value analysis value engineering (VAVE) workshop to determine the main attributes/requirements and their weighting of the lateral brace.

- Conducted an in-depth literature survey on attachment technologies for composites, with a focus on solutions for hybrid joints, effects of 3-dimensional (3D) reinforcement, bolted joints, and fatigue testing.
- Established a test matrix for coupon testing for baseline steel-steel joints, as well as the steel-composite system of interest.
- Selected commercially available, component-independent material and identified appropriate mechanical tests to investigate levels of damage resulting from various hole fabrication methods and bolt preload levels, as well as ways to mitigate the negative effects of the damage.
- Set up a vacuum infusion lab at Delphi Research Labs (DRL) to fabricate the initial test panels.
- Identified several software vendors as compatible with structural composite elements and interviewed them regarding their capabilities.
- Chose one commercial FEA software package to perform the structural analysis and design optimization and another to handle damage simulation and fatigue modeling.

Future Direction

- Continue FEA design and optimization, plaque testing, and fiber architecture determination in FY 2004.
- Conduct a requirements and concepts gate review to finalize the part geometry. Begin manufacturing the prototypes in 2005. In the second quarter of 2005, choose unique applications for both original equipment manufacturers (OEMs), followed by VAVE workshops and FEA and design optimization.
- In the last quarter of 2005, conduct the design and development gate review and initiate production part building.
- Optimize the final design, which will be submitted to track testing during the last quarter of 2006.
- By 2007, finalize the manufacturing process, supply product prints, and validate the product. PPAP will occur in the first quarter of 2007.
- Follow a similar approach for the primary beams project, beginning in the third quarter of 2005.

Introduction

The purpose of this project is to lead the rapid implementation of lightweight composite materials in Class 7/Class 8 vehicles via the development of advanced composite support structures, including chassis lateral braces and primary beams. Mass reductions are targeted for 50%. The benefits of mass reduction in commercial vehicle applications are well known. They include increased fuel economy and larger payload, which translates into fewer total trips and therefore fewer vehicles on the road. This leads to less traffic, which aids highway safety and decreases emissions. Support structures offer an opportunity for significant weight savings. However, this area of the vehicle also represents a large hurdle in terms of composite applications and market acceptance.

Composite lessons learned from previous industry primary beam studies and the technological achievements gained during the lateral brace study, when applied to the primary beam study, should result in a potential mass reduction exceeding 100 kg for a typical Class 8 tractor.

Our team is resolved to develop fiber-reinforced-composite technologies for these structural applications while determining the extent of their *durability*. In this respect, 3D fiber architecture will be examined. Composites with fibers in the Z direction have shown improved ability to hold the in-plane fiber architecture together under high fatigue and creep. They also resist interlaminar crack propagation, which may lead to delamination. Attachment technology will be developed, including but not limited to preformed holes in the fabric before consolidation, metal plate inserts, and post-forming (e.g. drilling) operations. This joining technology will require development, but we expect it to be applicable for both lateral brace and primary beam composite attachments.

This work is being coordinated with "Attachment Techniques for Heavy Truck Composite Chassis Members," led by Oak Ridge and Pacific Northwest National Laboratories, to help resolve the joining challenges of this program.

VAVE Workshop Results

The VAVE workshop and its pre-event were successful in providing the necessary roadmap for this program. First, a current production lateral brace was chosen for the first phase of this program. Prints and design specifications were also provided. It is for a bulk hauler (heavy-duty) application.

The major attributes of this program were determined and weighted. Attribute weighting assumes that the minimum requirement has been satisfied for all attributes; hence it determines where additional effort should be focused after minimum requirements are satisfied. Cost factors emerged as the major attributes of concern—the top three attributes were cost related. The purchased cost premium, the actual cost penalty for mass savings, is the most important attribute.

Cost of assembly was the second-most-important attribute. The OEMs pointed out that a process that is very disruptive to their assembly lines would be difficult to integrate and would be expensive if new equipment and training had to be implemented.

Warranty (after-sale cost) was the third-ranking issue. An issue important to the immediate financial costs of warranties is that a new composite member cannot be returned from the field more often than its steel counterpart. Otherwise, the image of structural composite materials incorporated into a heavy-duty truck chassis could be tarnished for a long time to come.

Process robustness was found to be the fourth most important attribute. This actually represents the scrap rate of the product before it is sent to the assembly line.

Modeling

Modeling includes FEA, optimization, and prediction of damage, fatigue, and failure. Many software vendors were interviewed and evaluated for participation in this project. Our main criteria were

- Composite design-analysis tools to aid in finite element modeling of composites, such as the prediction of fiber angle orientation from a 2D fabric in a general 3D mold configuration (draping). Such tools exist and are composite-specific.
- Process simulation for composites.
- Modeling of composites with 3D fiber architecture.
- Modeling of joining of composite parts to other composite or metallic parts, such as in bolted connections or adhesive bonding.
- Nonlinear material modeling of composites, specifically for stiffness degradation and material failure.
- Durability assessment for composite structures.

A composite capability matrix of the major commercial FEA packages was developed. The comparison was limited to the codes available at Delphi. We believe that there will be a need to use more than one FEA code for the design analysis in this project, since it is unlikely to that one code can do it all or will be familiar to everyone. One commercial FEA software package was chosen to perform the structural analysis and design optimization, and another was chosen to handle damage simulation and fatigue modeling.

Joining and Material Characterization

Initial test specimens were fabricated using an E-glass/vinyl ester system. A 3D woven 72-oz fabric was chosen. For these tests, we used material with equal amounts of 0º and 90º glass fiber and a 1.7% by weight through-thickness z-fiber) reinforcement.

The first four plaques created used a glass plate as a bed and a vacuum bag as the opposite surface. The samples fabricated had noticeable dimples on the vacuum bag side surface. These dimples were created where the z-reinforcement stitching went through the material. The glass-side surface was smooth. The dimples could create sites for crack nucleation, which would not be present in a two-sided resin transfer mold. In order to create samples with a more uniform surface, a tempered glass plate was used as a top plate and then the vacuum bag material was placed over the glass. The plaques created using the glass plate had smooth surfaces and were thought to be much more representative of an eventual production part.

Tensile testing has been performed using 0º, 90º, and 45° samples. The 0º direction is in the direction of the fabric as it comes off the roll; 90º is across the roll. The samples were tested according to ISO 527-4 (1mm/min). Presently, E_{12} is calculated based on an assumed Poisson ratio obtained for a similar E-glass/vinyl ester system from another project. The Poisson ratio will be measured pending repair of the appropriate equipment.

A neat resin plaque was created and cut into six "dog bone" specimens 75 mm long with a gage section of 5.1 × 3.0 mm. They were tested at a rate of 1mm/min. Five of the six samples yielded usable data.

These test results will be used as inputs for the FEA software. DRL began initial damage simulation/fatigue software training the week of November 17. The first model to be analyzed using the damage simulation/fatigue package will be a tensile bar. The software has a library of material characteristics that will enable a "first pass" analysis of a component. We will compare this initial analysis with the actual data. This comparison will give us a confidence level when using the damage simulation/fatigue software to evaluate a material system for which we do not have samples readily available for testing. The data will also enable tuning of the software to our composite system.

Conclusions

This project has been successful in identifying an application and receiving appropriate input from its members to correctly determine areas of focus for its study. Additionally, the format for determining which software to use for process and performance modeling (including fatigue and fracture) will prove to be valuable as the program continues. Coupling the efforts of this program with the project "Attachment Techniques for Heavy Truck Composite Chassis Members" has already shown to be invaluable in efficiently gaining progress on our goals. We are close to our timeline and anticipate continued success as we dive deeper into fabricating and testing parts and applying our modeling techniques.

F. Hybrid Composite Materials for Weight-Critical Structures

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RLO1830

Objective

- Develop and demonstrate (1) the application of hybrid composites and composite/metal hybrids to heavy-duty vehicles and (2) the ability of these material choices to be integrated into moderate-volume production.
  - — Develop and demonstrate the potential for major weight savings (>50% on a component basis) in critical structures applicable to truck cabs and support components.
  - — Demonstrate via proof-of-principle experimentation the basis for use of hybrid metal-composite systems to reduce weight.

Approach

- Investigate the potential of new materials and manufacturing technologies to effect major weight reductions for heavy-duty vehicles.
- Assist in demonstrating the applicability of composites and composite/metal hybrids to operational vehicles with little or no cost impact.
- Provide the experience base to develop the design and analysis tools, as well as the scientific understanding of the factors affecting molding and materials performance.
- Provide the materials suppliers with a market that can stimulate demand, leading to an increase in their production capacity. This will help reduce materials costs by creating higher volumes.

Accomplishments

- Completed the modeling of a door system for an existing PACCAR truck model and calibrated it against previous PACCAR models and experimental load/deflection data by Pacific Northwest National Laboratory (PNNL) staff.
- Ran a series of modified models simulating the addition of local carbon fiber stiffening strips. Developed an optimized reinforcement that provides a reduction in deflection of greater than 50%.
- Received the first sets of hybrid glass/carbon fiber preforms for liquid molding from the National Composites Center (Kettering, Ohio).
- Completed evaluation of test methods for the various hybrid materials.
- Provided several production doors from PACCAR to PNNL for application of the stiffening system. Prior to delivery of the doors, PACCAR performed baseline deflection tests that are currently being repeated with the carbon fiber stiffening strips applied.
- Began fabrication of prototype components for the hybrid door design by several vendors. The PACCAR/PNNL team recently completed the formal design review and design release meeting.

Future Direction

- Complete components for the hybrid door prototype demonstration during December 2003 and early January 2004.
- Complete the design of the door assembly fixtures and tools by PACCAR in preparation for prototype assembly.
- Complete assembly of three prototype hybrid doors by PACCAR and conduct truck cab structural testing.
- In conjunction with Mercia, update manufacturing cost models and review cost results for the hybrid door designs with PACCAR.
- Compile a final report at the conclusion of the prototype demonstration phase.

Introduction

Current materials and manufacturing technologies used for heavy vehicle door systems are often dictated by the high cost of tooling and the relatively low production volumes for Class 8 trucks. Automotive-style stamped door designs, whether of steel or aluminum, require multistage stamping dies that are generally cost-prohibitive at lower production volumes (<50,000 units per year). Alternate materials, such as glass-reinforced sheet molding compound (SMC), require less expensive tooling and can provide Class A finish; but the relatively poor specific properties of SMC tend to compromise design and result in a heavier door system. For many production truck cabs, a simple aluminum extrusion frame is used with a flat aluminum sheet riveted to the frame. Although this approach does not require expensive tooling, the use of constant cross-section extrusions in the frame is less than optimum; and it requires more assembly labor than other approaches. PACCAR, a world leader in Class 8 truck design and manufacturing, teamed with PNNL to explore alternate “hybrid” door system designs that minimize tooling cost and per-part door cost, while providing a lightweight, structurally stiff, automotive-styled door.

Project Approach

The initial approach to development of the hybrid door system was to perform a structural analysis of an existing PACCAR door design and determine what the design and performance goals should be for new-generation door systems. PACCAR provided a number of weight, cost, and performance parameters that it considered important for future door designs. PNNL was tasked to survey existing and emerging materials and manufacturing approaches that could be applied to a new door design. Following completion of this survey and analysis of existing door designs, PNNL, with design assistance from Mercia, Ltd., developed a series of five door design concepts that included combinations of large die castings, extrusions, carbon- and glass-reinforced composites, and conventional SMC and stamped aluminum exterior panels. Following a concept review meeting with PACCAR, an optimized hybrid door design concept was selected. The door concept was then defined using computer-aided design tools and analyzed with finite element models to validate performance, weight, and cost. After determining that the prototype design met or exceeded all performance and projected cost targets, PNNL and PACCAR selected methods to produce prototype components for the full-scale assembly and testing phase of the project. The finite element model of the prototype door system is shown in Figure 1.

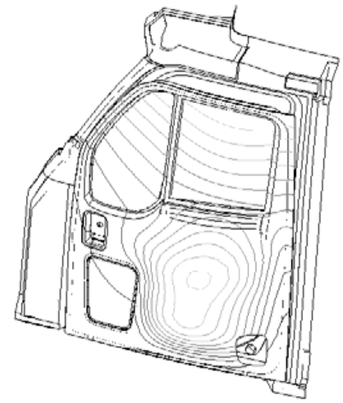

Figure 1. Finite element model of prototype door system under simulated loading conditions.

Conclusions

A door system has been designed and analyzed by the PNNL/PACCAR team. Development of three prototype doors for cab testing is currently under way. The hybrid door design provides attractive weight and cost savings compared with automotive-style stamped door designs, and it provides significantly reduced part count and assembly cost. Development of prototype door assemblies is currently under way. When complete, it will be subjected to a full range of cab durability testing by PACCAR.

G. Application of Carbon Fiber for Large Structural Components

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RLO1830

Objective

- Develop selective reinforcement technology that can be applied to large cab components to improve specific stiffness and strength while reducing overall component weight.

Approach

- Determine how well the low-cost carbon fibers can be hybridized with glass fibers to provide substantial weight and cost reductions in large cab components.
- Develop a system for preforming carbon and glass fibers together that will allow components to take maximum advantage of the capabilities of selective reinforcement alignment and property contribution.
- Develop models for the analysis of hybrid chopped-fiber preforms and composites that allow the thermal and structural properties to be developed and compared with experimental analysis.
- Perform structural testing to define the limits of applicability of the carbon/glass hybrid reinforcement materials to the large structures and develop guidelines for applications that may be used by original equipment manufacturers (OEMs).
- Design and develop critical subsection components of large structures to use in correlation with the predictive models, and validate the structural application criteria. Determine the capability to fabricate the materials in full-scale components, and determine the performance of these full-scale components in real application scenarios.

Accomplishments

- Developed short-fiber composite models for predicting properties of hybrid composite structures.
- Completed experimental testing of hybrid composite panels for model correlations.
- Developed a method to measure fiber angles for determining the level of anisotropy in chopped-fiber composites.
- Completed a set of designs of experiments on a new resin system for use with the hybrid composites.

- Completed initial runs on a large test tool with short-fiber composite preforms.
- Developed a large-scale subcomponent tool specifically to mimic design attributes applicable to several structural and appearance components.
- Performed initial runs on a large test tool with short-fiber composite preforms at a tier-one supplier.
- Developed hybrid fiber preforms for a test tool and demonstrated initial successful molding with hybrid fiber and resin system.
- Prepared samples for testing from large structural components.

Future Direction

- Scale up to test production parts.
- Continue testing new hybrid resin system.
- Develop modified tooling to be compatible with processing rates and materials selection for large hybrid structures.
- Analyze and experimentally verify techniques developed for improving appearance on components to demonstrate applicability to hybrid system.
- Verify tooling on bench and component scale parts.

Introduction

Selective reinforcements with higher-stiffness fibers have the potential to reduce both costs and weight simultaneously even at today's market costs. Their introduction is hindered by a lack of understanding of the fibers in existing processes, as well as the need to develop robust methods of preforming glass and carbon fiber materials together. In addition, the capability to meet Class A surface finish specifications is required, which requires potential development of bonding agents as well as the ability to model thermal and structural performance of the materials in a hybrid system. The recent development of composite systems combining carbon fiber reinforcement with low-cost automotive and marine resin systems provides the opportunity for selective reinforcement of a broad range of structural composite components.

The purpose of this project is to develop the design and materials processing technology to facilitate the application of low-cost hybrid glass fiber and carbon fiber reinforcements on large composite components, resulting in reduced weight and improved structural performance. The project will also seek to advance low-cost carbon fiber materials developed by industry by advancing the introduction of low-cost resin systems that are compatible with current heavy vehicle structural composites.

Predictive Modeling

Summary of the Modeling Approach

A computational tool called the Eshelby-Mori-Tanaka approach (EMTA) has been developed to predict the elastic and thermal properties (thermal conductivity, thermal expansion coefficients) as well as the elastic-plastic responses for short-fiber composites.[1] The EMTA model makes use of the Eshelby equivalent inclusion method in which fiber characteristics (including shape) are accounted for through the so-called Eshelby tensor while the constituents' properties, volume fractions, and fiber interactions are taken into account through the homogenization procedure. EMTA can handle two types of fibers mixed and embedded together in a resin matrix (hybrid composites). Planar orientation is assumed and obeys the fiber orientation distribution density of the form: $\rho(\theta) = \lambda e^{-\lambda\theta}$, where θ is the orientation angle measured with respect to the major fiber alignment axis (or fiber orientation distribution axis), and λ denotes a parameter governing the randomness. When θ is very small ($\lambda \to 0$), the fibers are randomly oriented; they are aligned for large values of λ. Between these two limiting cases, a given value of λ designates a fiber orientation distribution, which can be rather random or rather semi-oriented. EMTA can be used as a standalone code to predict the basic homogenized composite properties. It has also been introduced into ABAQUS by means of the user subroutine UMAT of this code for structural analyses.

Unsymmetrical Hybrid Composite Laminates

Unsymmetrical hybrid composites made of different composite layers, which are not symmetrical through the thickness, are of particular concern because of the mismatch of coefficients of thermal expansion (CTE) between the layers, which can lead to unacceptable structural deformations. On the other hand, adjusting the CTE by means of trial-and-error approaches is time consuming and expensive. Therefore, numerical methods to predict the CTE of the composite layers and the governing microstructural parameters (fiber volume fraction, aspect ratio, orientation distribution, etc.) are very helpful in assisting the design of unsymmetrical hybrid composite laminates.

The following discussion illustrates the procedure based on the EMTA for adjusting the CTEs for a hybrid laminate made of carbon/vinyl ester and glass/vinyl ester (hydrex 100) layers. First, an initial molded hybrid panel without adjustment of fiber volume fractions or orientations was analyzed. The panel dimensions are 610 mm × 610 mm × 3.302 mm. The EMTA thermoelastic model implemented in ABAQUS was used to determine the warpage of this panel due to its cooling from the curing temperature (65.55ºC) to the room temperature (24ºC). The comparison of the predicted and experimental deformed configurations serves to validate the modeling results. The mismatch of CTE is then remedied by adjusting the fiber orientation distribution and volume fractions. The CTEs of the constituent materials are given in Table 1. The CTEs of the glass and carbon tows filled with vinyl ester were predicted using the EMT model (provided in Table 1).

Table 1. Thermal expansion coefficients of the constituent materials and the fiber tows filled with hydrex 100.

	α_1 (×E6/°C)	α_2 (×E-6/°C)	α_3 (×E-6/°C)
Carbon[a]	-0.7	10	10
E-glass[a]	5.4	5.4	5.4
Vinyl ester[b]	70	70	70
Carbon/vinyl ester[c]	-0.011935	38	38
Glass/vinyl ester[c]	7.7081	36.525	36.525

a Source: *Handbook of Composites*.[2]
b Based on the values of thermoset polyester given in *Handbook of Composites*.[2]
c EMTA prediction.

The parameter λ, which characterizes the fiber orientation distribution, is about 0 for the glass layer and 1.1 for the carbon layer. Hence, glass fiber orientations are more random than those of the carbon fibers. Figure 1 shows the predicted deflection (displacement u_3) resulting mainly from the mismatch of the in-plane CTEs (α_1, α_2) between the carbon and glass layers. The predicted deformed shape agrees well with the experimental one shown in Figure 2.

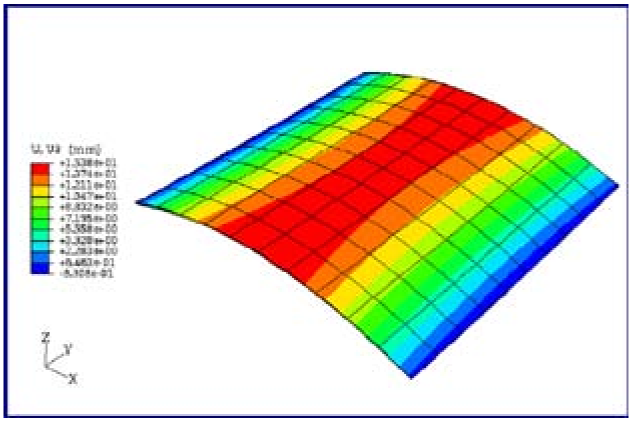

Figure 1. Prediction of the deformed shape and out-of-plane deflections for the analyzed hybrid panel after molding.

Figure 2. Warpage of the hybrid panel after molding.

Predicted and experimental deformed configurations are presented in Figure 3 in terms of the spatial coordinates *X*, *Y*, and *Z*. In both configurations, the *Z* coordinates were determined on the top surface of the hybrid panel. The predicted values correlate well with the experimental results.

To reduce the mismatch of CTE, the first remedy is to realize the same orientation distribution for the carbon and glass fiber tows. If the carbon tows' orientations are as completely random as those of the glass tows for $\lambda \rightarrow 0$, such orientations will lead to the

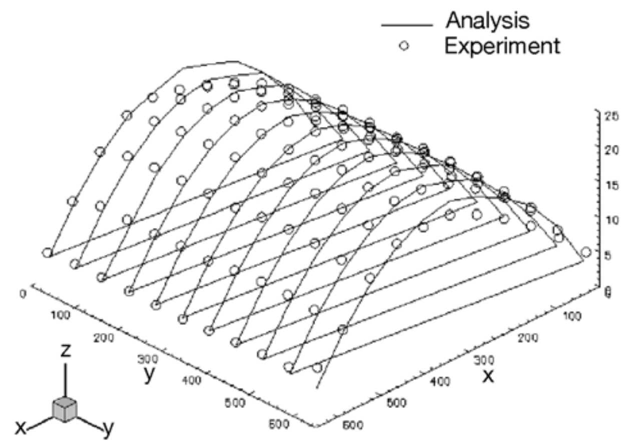

Figure 3. Experimental and predicted deformed coordinates for the analyzed hybrid panel after molding.

predicted variations of the in-plane CTE as a function of the fiber tow volume fraction shown in Figure 4. For a given glass tow volume fraction, these curves provide the corresponding carbon tow volume fraction, which must be realized so that the mismatch of CTEs is zero. In this example, the glass tow volume fraction is fixed at 0.5155 while the carbon tow volume fraction is initially 0.6771. These values were obtained knowing the volumes and the fiber volume fractions of the glass layer and of the carbon layer as well as the fiber volume fractions of the tows. In order to realize the same in-plane CTE for glass and carbon layers, the carbon tow volume fraction must be adjusted at 0.42, which gives rise to the carbon volume fraction of 0.27 in the carbon layer. Finally, Figure 5 shows the simulation results for the adjusted case. The deflection is practically reduced to zero as the result of the suitable adjustments of fiber orientations and the carbon tow volume fraction in the carbon layer.

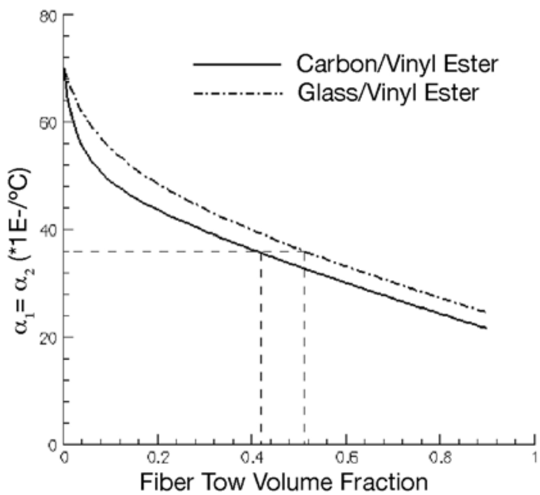

Figure 4. Variations of the in-plane CTEs of the carbon and glass layers with the fiber tow volume fraction.

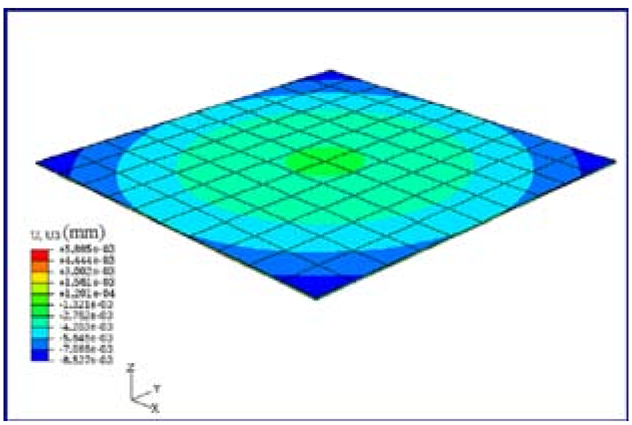

Figure 5. Prediction of the deformed shape and out-of-plane deflection for the analyzed hybrid panel after adjustments of fiber orientation distribution and fiber volume fraction for the carbon layer.

Experimental Testing

As previously mentioned, test panels were fabricated from preforms created by the National Composites Center (NCC). Figure 6 shows a typical carbon/glass hybrid panel. The panels had varied two target levels of volume fraction of glass and/or carbon. The hybrid panels also had three levels of carbon fiber in addition to the glass. Carbon volume levels were 3%, 9%, 15%, and 45%; the

Figure 6. Typical carbon/glass hybrid panel.

balance were random glass, with the exception of the 45%. These were made in two nominal thicknesses of 2.5 mm and 4.0 mm. There were also fiber-directed preforms of carbon fiber for comparing how well the properties could be controlled for a specific orientation. The panels were then resin transfer molded (RTM) with a standard polyester RTM resin from Reichhold Corp.

The panels were then laid out for specimen orientation and cutouts. The specimens were oriented with one referenced edge of the panel, and specimens were then removed with one set being parallel (0%) and the other being perpendicular (90%) to the referenced edge.

Tensile and flexural specimen geometry followed the ASTM D638 Type I Tensile and ASTM D790 Four-Point Bending Standards, respectively. Specimen profile measurements were taken and recorded, including width and thickness for each specimen. The specimens were also asymmetric in the panel through thickness.

The tensile specimens were bonded glass-to-glass with the carbon layers on the outer surfaces, then cut to the specification geometry. The double bonding was used to avoid bending moments during testing. Double extensometers were then used to measure the axial elongation and the width strain during axial loading. From this double extensometer measurement, a three-dimensional strain response can be calculated. These data are then used to develop the elastic stiffness matrix and constitutive equations for model development. The flexural specimens were tested both with the carbon side up in compression and down in tensile for four-point bending. This allowed the modeler to be able to compare how well the prediction correlated with experimental data.

Fiber orientation measurements were made for determining the randomness factor for the EMTA model. Figure 7 shows a 3% carbon fiber preform that was used to determine the random pattern of the chopped material applied to the preform.

Figure 7. Composite hybrid preform with 3% carbon fiber chop.

The preform digital image was then used in a software package from which fiber orientation was measured relative to the bottom edge. The orientation was from 0 to 180º. Figure 8 shows the output data from the Figure 7 panel. The data show very good randomness with no defining pattern. NCC also provided some fiber-directed preforms for evaluation. The panels were to have 0 and 90% directed fibers. Figure 9 shows the fiber-directed panel from which data was collected. These measurements proved to be more challenging, with the dark background and little contrast. The lighting was extremely important for enhancing the fiber tows on the surface.

Figure 10 shows the histogram data of the Figure 9 measurements. A pattern is developed by the preferential orientation of

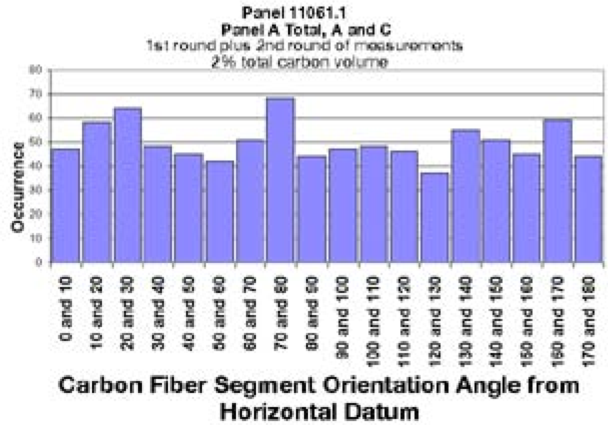

Figure 8. Histogram of fiber orientation.

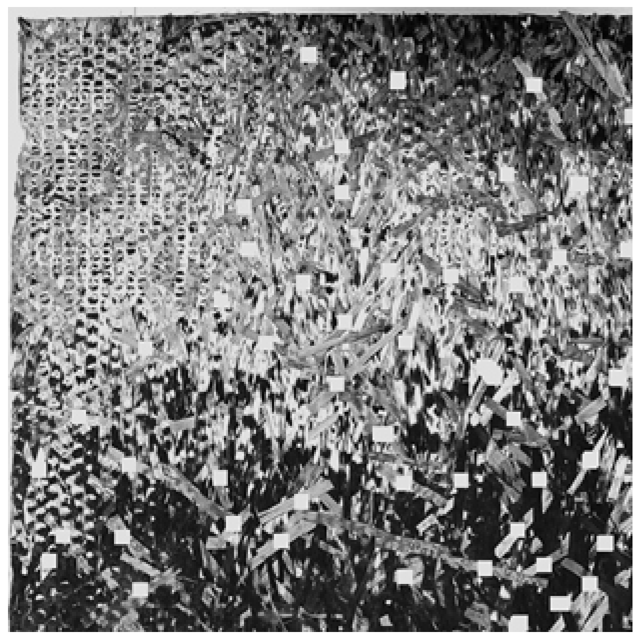

Figure 9. Fiber-directed panel.

the chopped fiber pattern program from the chopper gun. The histogram shows a bimodal distribution of fibers at approximately +45 and 60%.

Model and Experimental Correlation

A representative region of the specimens was used and discretized in three-dimensional finite elements for the simulation of the tensile stress/strain

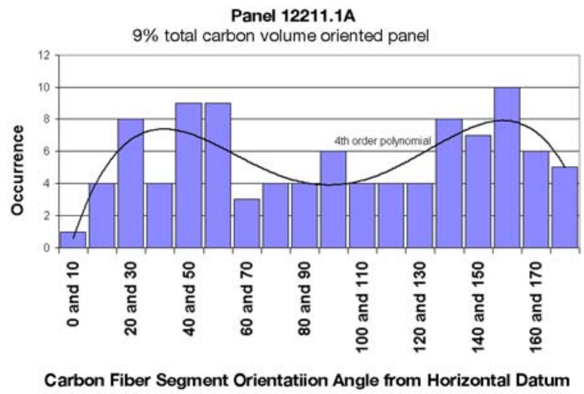

Figure 10. Histogram showing bimodal distribution of carbon fiber.

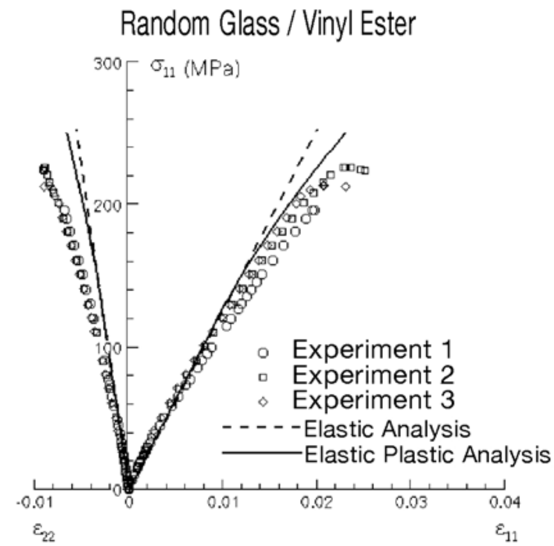

Figure 11. Tensile stress/strain responses of the random glass/vinyl ester specimens.

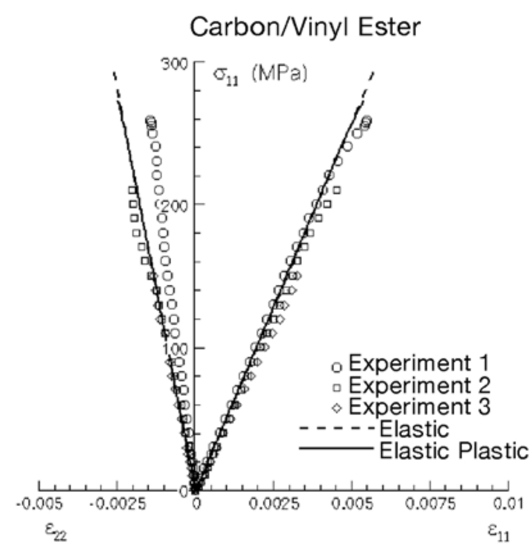

Figure 12. Tensile stress/strain responses of the random carbon/vinyl ester specimens.

responses. This region is 13 mm wide (actual specimen width)and 26 mm long. The average thicknesses of the glass and carbon specimens are 4.7 and 5.5 mm, respectively. The major fiber alignment axis is parallel to the loading direction. Figures 11 and 12 present the simulated tensile stress/strain responses compared with the experimental results for the glass/vinyl ester and carbon/vinyl ester specimens tested until failure. The simulated curves were obtained using the elastic EMTA model (dashed line) and the incremental elastic-plastic EMTA model (solid line) implemented in ABAQUS. The glass/vinyl ester specimens could undergo higher deformations than the carbon/vinyl ester ones. In the former case (Figure 11), after a linear behavior at small strains, the responses become nonlinear at higher applied stresses; in the latter case, the nonlinearity of the responses is negligible. The nonlinearity results mainly from two different material origins. The first origin is damage by matrix cracking, fiber/matrix decohesion, and fiber pullout and breakage at the final stage. The second cause results from the plastic deformations of the matrix material. In this report, damage was not modeled, and only the plasticity of the polymer matrix was accounted for.

Figures 11 and 12 show good correlations of the predicted results with the experimental values except around the final loading stage, where excessive accumulations of damage led to failure of the specimens.

Flexural tests (based on the ASTM D790 standards) were conducted for glass/vinyl ester, carbon/vinyl ester, and carbon/glass/vinyl ester specimens. The latter are made of two layers—one carbon/vinyl ester and one glass/vinyl ester layer. In all specimens, the major fiber alignment axis is parallel to the width direction. Figures 13 and 14 show the predicted load/deflection curves compared with the experimental values also presented on the same figures for the glass/vinyl ester and carbon/vinyl ester specimens. In all cases, the vertical deflection was determined at the center of the bottom surface of the specimen. Good correlations with the experimental results were obtained with the use of the elastic-plastic EMTA model. It is noted that excessive damage leading to final failure caused the load to drop at the end of the loading. Prior to final rupture, the elastic-plastic solutions predict the load/displacement responses very well. An elastic-plastic analysis using the large displacement (geometric nonlinearity)option in ABAQUS was also carried out in order to determine the effect of large displacements, which

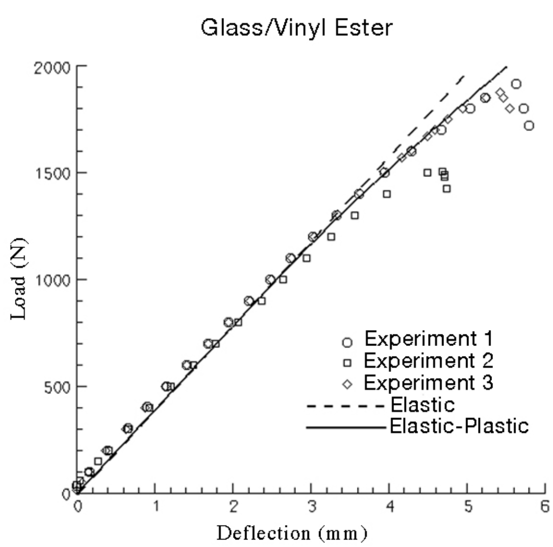

Figure 13. Flexural load/deflection responses of the random glass/vinyl ester specimens subjected to four-point bending.

occur around the end of the loading, on the overall responses. The corresponding solution for the carbon/vinyl ester specimen is denoted as "NLG Elastic-Plastic" and is presented in Figure 14. Compared with the elastic-plastic solution of the same problem,

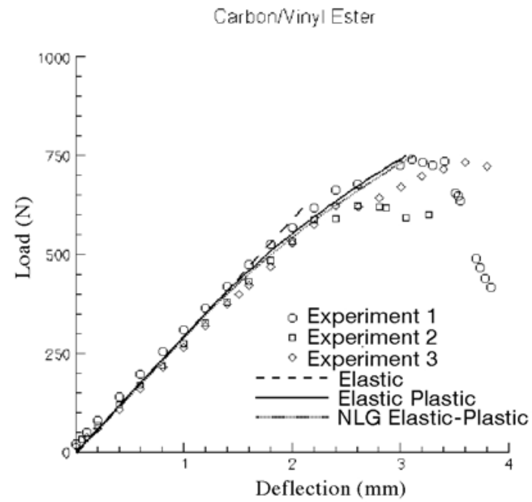

Figure 14. Flexural load/deflection responses of the random carbon/vinyl ester specimens subjected to four-point bending.

it is noted that the geometric nonlinearity is negligible. This is because the maximum deflection is of the same order of magnitude as the specimen thickness. Therefore, it is not necessary to carry out elastic-plastic analyses accounting for geometric nonlinearity for these specimens.

The predicted and experimental load/displacement responses for the four-point bending carbon/glass/vinyl ester specimens are presented in Figure 15. The test configuration was such that the carbon/vinyl ester layer is the upper layer. Figure 15 shows a noticeable scatter in the experimental results, especially at high loading levels leading to failure. The predicted elastic-plastic curve deviates from the elastic one when the deflection has attained 1.4 mm. It is also noted that early occurrence of damage has led to failure of these specimens at relative low load levels as observed in the carbon/vinyl ester specimens (Figure 14).

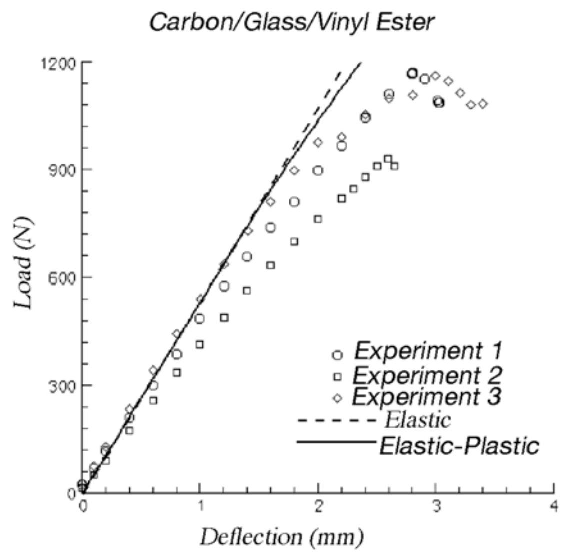

Figure 15. Flexural load/deflection responses of the random carbon/vinyl ester specimens subjected to four-point bending.

Hybrid Resin Evaluation

A Minitab design of experiments was run using a full factorial design with two levels and three factors. The interests in the experiments were to help determine which components gave the most control over the gel time. The factors of interest were temperature, cobalt content, and catalyst. The output measurements were gel time, peak exotherm time, and peak temperature.

The experiments were run on a Haake Rheocord 90 mixer using Brabender medium-shear mixing blades. The new hybrid resin from Reichhold was used. The catalyst, cobalt promoter, and temperature were controlled for the experiments.

The data were collected based on time, temperature of the resin, and the torque value during the mix. The gel time was taken as the first inflection of the torque curve. This is based on the torque value beginning to climb during resin gelling. The resin temperature also begins to increase relative to the same point as the gel time. The peak exotherm time is taken at the peak temperature that the mixture reaches before cooling down.

Figure 16 shows a Paretto chart that illustrates the standardized effects from the experiments. The Minitab output compares the different factors and combinations of effects. The Paretto chart clearly indicates that temperature has the most effect on gel time, followed by the cobalt content. The combined effect of cobalt and temperature also is a key contributor to the gel time.

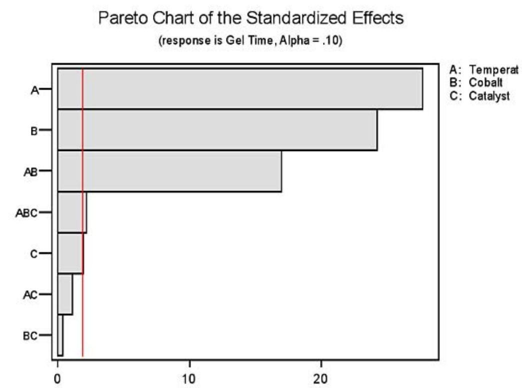

Figure 16. Paretto chart for gel time.

The catalyst showed very little effect on the gel time, and the trend was for the lower catalyst content to trend higher in peak exotherm temperature. However, the difference in peak temperatures is not statistically valid and is not being considered a concern.

Preform Evaluation

Several different preform styles have been considered and evaluated for the program to date, including commercial and development variations to achieve the goals for fiber hybridization. Using the DOE/Automotive Composites Consortium P4 preforming cell at the National Composites Center, hybrid flat preforms were made in a continuous process by modifying equipment to meet requirements. This was a distinctly different approach to the previous efforts where separate preforms of carbon and glass were made and later bonded together by NCC. Issues were seen in the distribution of binder in this approach, especially given the preferred flat tow form of carbon fiber being used. Molding of these panels indicated a lack of binder and/or lack of fully developed strength for interlayer adhesion, especially in the glass fiber layer. Preforms were later developed for a multisided test tool, as well as for the large test mold used for full-scale evaluation trials. These confirmed the lack of development of binder strength at the interface, because the all-carbon preforms molded extremely well, and the glass preforms showed issues with fiber washout and lack of preform integrity.

A preform fabrication cell was developed to allow rapid changes to the preform structure and subsequent molding trials. This allows evaluation of specific approaches to be made more efficiently and cost-effectively. Hybrid preforms were developed that have given complete mold filling and wetout capability and provided samples for mechanical property testing. Work is continuing to make this process repeatable and predictable.

Preforming development continues on two fronts. Small tests are preformed for both the flat glass mold and multifaceted test tools for determining permeability and resin/preform interactions, and large preforms are made for evaluation of scale-up factors and the ability to meet design feature performance for subcomponent test tooling.

Large Evaluation Tool

In order to evaluate the capability of the process and materials to meet the demands of industry, a proof test component tool was developed. This tool has all the features anticipated in designs, and it was used to evaluate the hybrid process for achieving performance given the requirements of these features. It is not expected that all features may be molded and perform as expected. The goal of the development is to determine the limitations of the hybrid fiber system and, provide designers and OEMs with guidelines for which lightweight materials will be applicable, and to provide information on methods to design and manufacture these fibers.

Project personnel initially ran the evaluation tool over a week-long period at a tier one supplier. Figure 17 illustrates the size of the test tool for evaluating complex preforms and mold filling. It was immediately apparent that this was not a desirable development process because the systems for preforming, resin transfer molding, and resin optimization had to be developed independently and then brought in to the test. Limited capability to modify preforms and resin systems was achievable, given that the on-site capabilities were not necessarily geared toward development programs.

Results from the trials showed problems with the binder capability of the preforms, and the interaction of the resin system softened the binder over time. (This was not observed to this extent in smaller-scale test tools because the resin had much lower contact times with the preforms before the smaller tools were completely filled and the flow stopped.) All-carbon fiber preforms were much more successful than glass fiber preforms, and several test sections were useful for fabrication of coupons, etc. Indications from the test were that resin temperature and tool temperature control were critical for this system. This led to the work on the resin development experiments. Mold temperature was seen to be a significant issue, which has led to new approaches to tool design and build that are being evaluated.

A new cell was set up for R&D purposes with preforming modifications and resin modification possible in a more appropriate environment. Also, several options for molding were built in that were not possible in their existing production environment but could easily be implemented once proven. Running this cell, several successful test panel runs were made late in the year that used hybrid preforms and met the requirements of the overall process. These

Figure 17. Mold cell for test tool setup for large-part molding.

have provided samples to be tested early in the next phase. Development of parameters has been much more controllable; and several advanced options for preforming, molding, and materials are being investigated.

Based on initial results, the potential to meet industry requirements is very promising for the process. Researchers expect to achieve this capability on a scale that will provide confidence to adopt this lightweight materials technology. Figure 18 illustrates a successful hybrid carbon/glass composite from the test tool in Figure 17.

Conclusions

- In conclusion, a wealth of information has been gained from the tier one molding trials and the design of experiments on resin systems. The molding trials show that preform loft and strength need more fine-tuning. Carbon fiber preforms operation was especially promising; however, the initial glass fiber performance was disappointing, especially compared with lab trials. The tooling needs to have better temperature and resin flow control. Resin heaters for the two components will help in

Figure 18. A successful carbon/glass hybrid composite part from the large test tool.

viscosity and the addition of fillers in the future. Molding parameters will have to be addressed in a much more rigorous manner than this industry has been accustomed to, and statistical process control will become more of a norm to achieve consistent results. Samples of large-scale, as-molded hybrid fiber components are now available for testing and optimization. These have shown a significant increase in capability to the industry regarding potential component weight reduction.

- The design of experiments confirms that gelling was an issue for tier one suppliers and that researchers should be able to adjust the fill points based on the fill time-to-gel. The design of experiments indicates (1) the importance of temperature control on the tooling and cobalt content in the resin for completion of the fill and (2) the need to optimize cycle time to increase throughput for production cycles.

- A computational tool using the EMTA has been developed for the thermo-elastic and elastic-plastic analyses of short-fiber polymer composites. The tool was created by implementing the EMTA-based constitutive models into ABAQUS. The simulations of the tensile and flexural tests conducted on glass/vinyl ester, carbon/vinyl ester, and carbon/glass/vinyl ester specimens have provided good predictions of the composite responses, which were in agreement with the experimental results.

- The computational tool can be used to determine the basic properties needed for the design of short-fiber polymer composites, such as effective thermal and elastic properties, and tensile and flexural moduli of the composite laminate. Moreover, the ability to account for plastic deformations using the incremental elastic-plastic EMT model enables the composite behavior to be modeled at high loading levels, leading to deformations that exceed the elastic limit. The model inputs are fiber (or fiber tow) and matrix properties, fiber (or fiber tow) volume fractions, aspect ratios, and the orientation parameter (λ). This computational tool offers the flexibility to adjust these parameters so that the resulting homogenized properties and responses satisfy the design criteria in terms of desired properties and deformation limits. The tool can therefore be used to assist in the design and manufacturing of short-fiber composite structures.

- The design of experiments confirms that gelling was an issue for tier one suppliers and that suppliers should be able to adjust the fill points based on the fill time to gel. The design of experiments indicates the importance of (1) temperature control on tooling (2) cobalt content in the resin for completion of the fill and (3) cycle time optimization for increased throughput for production cycles.

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H. High-Conductivity Carbon Foam for Thermal Management in Heavy Vehicles

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objective

- Develop an understanding of the heat rejection demands of heavy vehicles and develop designs that utilize the superior performance of high-thermal-conductivity carbon foams.
- Coordinate efforts with a heavy vehicle manufacturer, an engine manufacturer, and a heat exchanger manufacturer in order to target the designs toward their understanding of the new vehicles' heat loads.

Approach

- Study fundamental mechanisms of heat transfer using carbon foam.
- Develop a testing method to evaluate foams and designs using the foams.
- Work with industrial partners to develop a complete understanding of heavy-vehicle-related issues.

Accomplishments

- Published a report summarizing the durability of graphite foam under simulated typical operating conditions.
- Constructed a cost model to determine the largest cost factors in the manufacture of graphite foam
- Developed a heat transfer model. Researchers determined that pressure drop across the foam is proportional to the flow length of the foam. In addition, permeability of the foam plays a role in the thermal performance.

Future Direction

- Continue collaboration with the Georgia Tech Research Institute (GTRI) on sub-scale and possibly full-scale airfoil heat exchangers.
- Evaluate potential on-vehicle tests to be performed at partner's facilities

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Introduction

A novel technique for creating pitch-based carbon foam was developed at Oak Ridge National Laboratory (ORNL)[1,2] before 1997. This technique uses mesophase pitch as a starting material but does not require the costly blowing or stabilization steps seen with typical carbon foams.[3]

The ORNL foam is an open-cell structure with highly aligned graphitic ligaments; studies have shown the typical interlayer spacing (d002) to be 0.3356 nm, very near that of perfect graphite (0.3354 nm). As a result of its near-perfect structure, thermal conductivities along the ligament are calculated to be approximately 1700 W/m•K, with bulk conductivities 180 W/m•K. Furthermore, the material exhibits low densities (0.25–0.6 g/cm^3) such that the specific thermal conductivity is approximately four to five times greater than that of copper. The very high surface area (20,000 m^2/m^3) combined with the high thermal conductivity suggests that graphite foam has significant potential for application as a thermal management material.

One issue of importance with regard to the performance of the ORNL foam is durability in typical operations. It is hoped that by understanding how the foam behaves in environments approximating actual operating parameters, researchers will learn how to modify the foam as necessary to meet application-specific needs. To this end, several durability studies have been undertaken, the constraints directed by either the research team or by industrial partners. The studies include thermal cycling, compression testing, corrosion, erosion, vibration, and salt spray (fog). This study was completed with joint funding from OATT and HSWR.

Results

Thermal cycling

Determining the effect of thermal cycling on the properties of graphite foam is important for the advancement of certain applications in the automotive industry. Graphite foam has a very low compressive modulus (0.08–0.114 GPa) compared with other materials commonly used in thermal cycling applications, such as aluminum (70 GPa). This low modulus value is expected to make graphite foam highly shock-resistant. Thermal cycling testing was carried out in two stages. The first stage was material durability testing, intended to determine whether the graphite foam itself could survive thermal cycling. The second stage, interface testing, was intended to study the effect of thermal cycling on the interface between the flat tubes and the foam on a subscale heat exchanger.

In the material durability test, foam blocks, press-fit with tubes, were tested for heat loss at the following intervals: 0, 3670, 6833, 16150, 26150, 36150, and 46150 cycles. The testing was accomplished by installing a foam block into a heat exchanger rig in which hot water was passed through the tubes and cooling air was blown over the foam block. The heat loss, or heat removal, was measured and plotted as shown in Figure 1. The figure shows a plot from the copper tube set, which is representative of the other three-tube material sets. A slight decrease in properties was found in the earliest cycles; then a plateau was exhibited and no further statistical changes were seen.

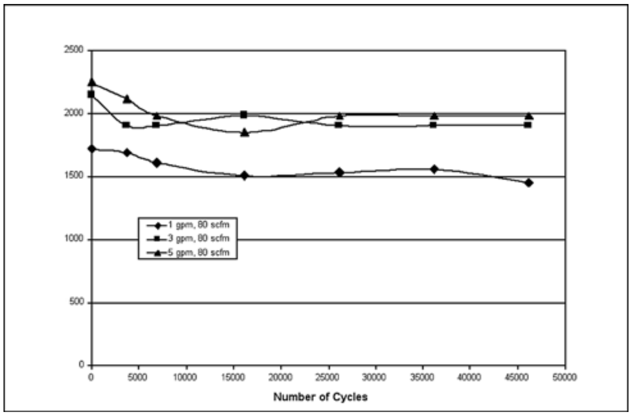

Figure 1. Water heat loss vs cycles.

This reveals that no significant degradation of the foam occurs foam as a result of thermal cycling.

In the sub-scale heat exchanger test, the bonding interface was visually inspected after every 4 h of testing. Visual inspection showed no apparent degradation of the interface. Even though visual inspection does not give a quantitative measurement of the status of the interface, this is an initial attempt to study this effect. Future efforts will focus on developing the means to quantify this property as a function of heat transfer.

Compression testing

Compression testing normally is conducted using a cylinder of a given ratio of length (*L*) to diameter (*D*). This ratio is typically between 1 and 3 (an L/D ratio of up to 10 may be used when very accurate measurements of the modulus are required). With larger ratios, the material being tested may buckle, giving rise to instability. With smaller ratios, end effects are more prevalent, resulting in barreling. Because brittle materials do not experience a significant amount of plastic deformation, a smaller ratio is used for these materials (typically 1.5 or 2). A ratio of 1 was chosen for the graphite foam, as it exhibits brittle failure and, furthermore, no deformation is observed; it appears that the foam fails locally in the ligaments, nearly a layer at a time. The tests were performed on 1-in. cubed samples of graphite foam to provide Young's modulus, compression strength, and strain to failure. These measurements are important in those applications in which it is necessary for the foam to exhibit some degree of structural integrity, for example, heavy-vehicle radiators.

After more than 42,000 thermal cycles (the same cycles used as in the thermal cycling tests), the graphite foam exhibited no statistical change in compressive stress (Figure 2) or compressive modulus (Figure 3). These results indicate that no structural changes or damage took place as a result of thermal cycling.

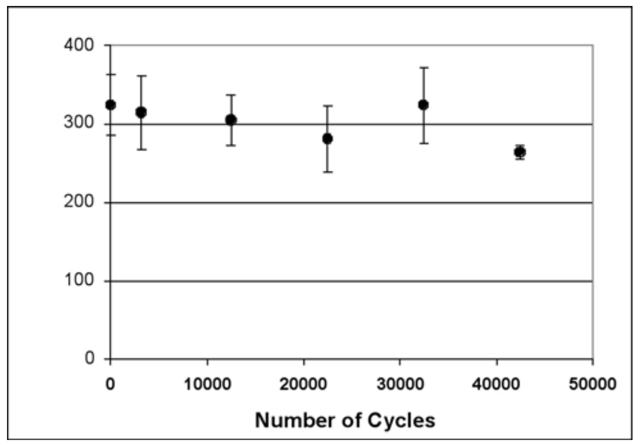

Figure 2. Graphite foam thermal cycling compression test results: maximum compression stress.

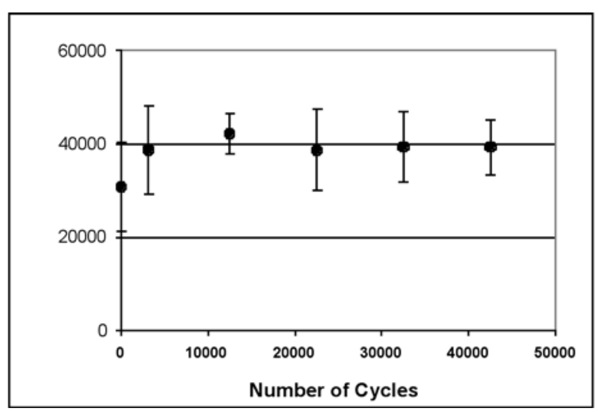

Figure 3. Graphite foam thermal cycling compression test results: compression modulus.

Graphite foam does not appear to be affected mechanically by thermal cycling. No effective decrease in properties was measured. Work is ongoing to verify that ASTM C 695 is indeed applicable to graphite foams; ruggedness testing was completed in summer 2003. Round-robin tests will begin in 2004.

Corrosion in propylene glycol/water mixtures

The use of graphite foam for power electronic applications has been proposed to dissipate heat in a variety of applications, including heat sinks, heat spreaders, and cooling inverters. In certain applications (e.g., a closed-loop heat sink that requires the use of a refrigerant), corrosion of the graphite foam in the presence of the evaporant or coolant becomes a concern—especially as it affects materials properties such as thermal conductivity, phase stability, and the reliability of a braze or weld. The reliability of the bond between the foam and a metallic substrate becomes increasingly important in an application such as a cooling inverter, in which no mechanical options are available for bonding the foam to the substrate. To address these issues, a corrosion study was completed.

A modified SAE J1211 standard test was used to expose samples of foam for 1000 h each at room temperature, 60°C, and 100°C in a 50% propylene glycol/50% water mixture (Zerex™). The samples consisted of untreated foam, nickel-coated foam, and nickel/polyvinylpryllidone (PVP)-coated foam. These samples were bonded to aluminum, copper, brass, and stainless steel plates using 50/50 (tin/lead) solder. In addition, unbonded samples of untreated foam, nickel-coated foam and nickel/PVP-coated foam were exposed at the same conditions to provide a base material control. After 1000 h of exposure at each of the three temperatures, each sample joint was examined visually and with scanning electron microscopy (SEM). Thermal conductivity was also measured.

A summary of the effects of temperature, foam treatment (coated or uncoated), and substrate on thermal conductivity after exposure in Zerex is shown in Figure 4. These results serve only as trend indicators for the

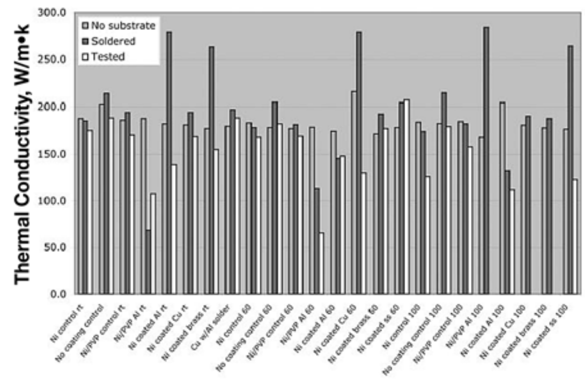

Figure 4. Effects of corrosion on thermal conductivity after 1000 h in Zerex solution at room temperature, 60°C, and 100°C.

feasibility of using this technique, as only one sample was tested for each condition. If the margin of experimental variation is calculated in the three control samples, the minimum variation seen even before testing is approximately 5%. Thus only variations above 5% were considered. The greatest effect upon thermal conductivity and viability of the solder was seen at the highest temperature condition, 100°C. Only one of the eight samples tested at 100°C showed no effect of temperature on thermal conductivity, and that sample was the uncoated control. Three of the eight samples tested at 100°C failed. Two of these samples—the nickel/PVP-coated foam soldered to an aluminum substrate and the nickel coated-foam sample bonded on to a brass substrate—failed at the solder or joint, i.e., the sample and substrate were found completely separated in the solution after exposure. The nickel-coated foam sample bonded to copper did not fail during testing (i.e., it was not separated upon removal after testing), but it separated upon being mounted into the fixture for measuring thermal conductivity.

It is evident even from this brief study that the foam structure itself is unaffected by exposure to Zerex at temperatures of up 100°C and times of 1000 h. However, thermal conductivity and the mechanical integrity of the solder attaching the substrate and foam will be a factor in the foam's ability to survive and the stability of its thermal conductivity values. This study is being reproduced using multiple samples for each condition to determine whether the observed trend at 100°C or any other trends are real. One sample, the uncoated foam sample, did survive the exposure to Zerex at 100°C with no effect on thermal conductivity. For some heat sink/power electronic applications in which only a mechanical bond is used, the result is very encouraging and shows that graphite foam is indeed a valid material for use. In addition, the variability of the thermal conductivity and survivability of nickel-coated foam samples at 100°C appears to depend upon the substrate/solder combinations. To address this effect, current work is examining different solders and substrate combinations that will also be included in future corrosion studies.

Erosion

The erosion tests performed on the graphite foam samples were designed to measure the mass loss and mass-loss rate at a given impinging air velocity. These tests will reveal the graphite foam material loss based on preparation procedures (e.g., coatings and other surface treatments).

Graphite foam specimens were machined from a billet of commercial graphite foam processed under documented conditions. Each erosion specimen was machined into a 1-in. cube with a 0.25-in. hole machined through in the center of the specimen. Each specimen was weighed to get the baseline weight prior to testing and then mounted in a fixture. The outlet air nozzle was mounted approximately 3 in. from the specimen, centered on the 0.25-in. hole. Air was blown onto the graphite foam specimen at an approximate velocity of 1600 ft/min face velocity for 5 min.

After 5 min, each graphite foam specimen was removed and weighed in order to get the post-test weight. The mass loss was reported by subtracting the post-test weight from the pre-test weight, and the mass-loss rate was reported by dividing this value by 5 min. The mass loss rate of the raw graphite foam was calculated to be 0.0049 g/min at a continuous velocity ,while the loss rate for the nickel-coated foam was calculated to be 0.0013 g/min at a continuous velocity.

The mass-loss rate exceeded acceptable limits set by industrial partners. While the nickel coating significantly decreased the mass-loss rate, the overall rate was still found to be unacceptable for any application in which the foam would be subjected to impinging airflow.

The test setup described has been modified significantly to accommodate higher velocities, including the use of an adjustable jig and various coatings (e.g., raw, copper-coated, nickel-coated, and silver-coated foam). Two samples of each coating were used, one with straight holes and one with rounded holes as stress concentrators. In future testing, each sample will be exposed to impinging air flow for 5 min, weighed precisely, exposed for 5 min more, weighed again, and so forth until either the sample catastrophically fails or a plateau is reached.

Vibration

Vibration testing of graphite foam mounted to a metallic substrate was conducted to provide information on the degradation within the graphite foam, as well as any debonding that may occur at the interface between the graphite foam and the metallic substrate. The response of the graphite foam specimens was dependent on the vibration test parameters. An industrial partner supplied the test parameters.

Graphite foam specimens evaluated by the vibration test were mounted to aluminum, copper, brass, and stainless steel sheet metal substrates to provide a structurally sound base for mounting the specimens to the vibration table. The specimens used for this test measured 0.625 in.[3] The different substrates were used to determine how the varying mechanical properties of the metals, mainly their damping characteristics, affected the response of the graphite foam to the given vibration test conditions.

The thermal conductivity of each specimen was measured, prior to the vibration testing, so that a direct comparison could be made following the testing, which would reveal degradation within the graphite foam and in the interface between the graphite foam and the metallic substrate. Eighteen specimens were evaluated in the test. None of the specimens showed any visual degradation or debonded from the metallic substrate. The thermal conductivity measured following the vibration test showed there had been no change compared with the thermal conductivity measured prior to testing. The lack of variation in the thermal conductivity, except experimental error, led to the conclusion that no structural degradation had occurred to the graphite foam ligaments that impart the high thermal conductivity of the graphite foam.

Salt spray (fog)

Graphite foam samples were tested by exposure to a salt fog to indicate the material's relative corrosion resistance under controlled test conditions. An independent laboratory, Environ Laboratories, LLC, located in Minneapolis, Minnesota, conducted the tests.

The full report from Environ Labs was prepared to ASTM standards and was received in July 2003.

ORNL measured changes in mass and thermal conductivity. These results serve only as indicators of feasibility or trend, as the instrumentation used during the thermal diffusivity measurements (from which the conductivity is calculated) was been called into question during this test period. The variability in the baseline conductivity data does not agree with prior data on this commercial foam; further, the variability is not as great in the tested data. Examination of the instrumentation is currently under way; further tests using joined samples are on hold until this issue is resolved.

GTRI Collaboration

Georgia Tech Research Institute has a patented aerodynamic heat exchanger (AHE), a high-lift airfoil section that incorporates a conventional radiator to provide heat transfer and cooling to an automobile (passenger or heavy vehicle). If flow is allowed through the airfoil, it can seriously degrade the aerodynamics of the system. Thus a study, jointly funded by OATT and OHVT, was undertaken to determine the effectiveness of ORNL's graphite foam in this novel airfoil design; the foam has excellent heat transfer characteristics while engineering designs may allow tailoring of the porosity.

Three AHEs were tested under the same conditions, a conventional radiator manufactured by Visteon (like the one installed in the GT MotorSports Formula SAE race car), a solid graphite foam core, and a corrugated graphite foam core. Each core had cooling tubing passing through internal channels (Figure 5), and for all configurations, the airfoil was mounted on a strain-gage balance below the floor to measure forces and moments. Data were taken first to determine the aerodynamic characteristics of each core by monitoring the slot blowing pressure at a constant angle of attack as well as the tunnel pressure. Then the thermal performance was measured

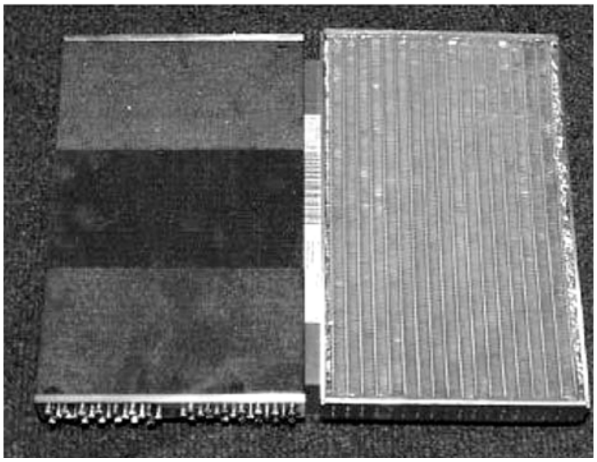

Figure 5. Solid graphite foam and corrugated foam radiator cores.

by having a constant coolant flow rate for variable pressure; further, the tunnel speed was also varied.

The heat transferred from the coolant can be expressed as

$$Q = \dot{m}_c C_p (T_{c_{IN}} - T_{c_{OUT}})$$

Measurements were made for each configuration at several freestream velocities, coolant flow rates, and blowing rates. Figure 5 shows the comparison of the graphite foam cores with the conventional Visteon core. Interestingly, the solid graphite foam core performed as well as the conventional core but with significantly less drag (Figure 6). Thus the dense graphite foam core has been shown to be an effective heat transfer medium that does not significantly affect the aerodynamic performance of the AHE.

Conclusions

The results from these studies are encouraging and indicate that the graphite foam performs well in most environments. Some issues have arisen with the choice of solder and/or substrate in certain

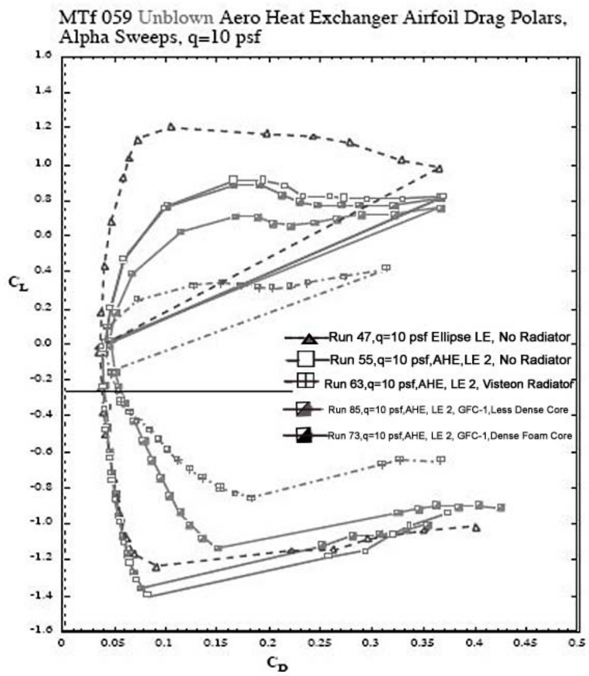

Figure 6. Unblown airfoil lift/drag polars as a function of angle of attack with and without radiators installed.

environments, and tests are ongoing to further optimize these systems.

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I. Lightweight Functional Composite Materials

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Objective

- Demonstrate adsorbed natural gas (ANG) storage densities at levels comparable to compressed natural gas storage but at significantly lower pressure and at comparable cost.

Approach

- Conduct research in six task areas:
  1. Process development and physical property characterization
  2. Materials characterization
  3. Modeling and simulation
  4. Prototype tank design construction and testing
  5. Vehicle testing
  6. Economic analysis

Accomplishments

- Made progress in analysis of selected samples from the ANG test system toward better understanding its performance. Developed a storage monolith capable of storing >180 V/V methane at 900 psi (September 2002).
- Characterized carbon materials using high-resolution transmission electron microscopy (TEM), scanning electron microscopy (SEM), small-angle neutron scattering (SANS), and inelastic neutron scattering (INS). Alternate carbon fibers and fiber precursors have been evaluated (March 2002).
- Initiated computer simulation work using a reverse Monte Carlo (RMC) approach.
- Performed a series of 12 experiments on samples machined from carbon monoliths selected from the Big Tank, along with additional tests using a modified experimental procedure.
- Completed the test and evaluation of the demonstration fuel tank (September 2002)

- Prepared a report on characterization of microporous adsorbent carbon-fiber-based monoliths made from alternate fibers (September 2002).
- Completed the design of an in-vehicle demonstration natural gas fuel tank (March 2002).

Future Directions

- Initiate vehicle testing in FY 2004.
- Initiate economic analysis in FY 2004.
- Determine why the storage monoliths used in the demonstration tanks did not perform at the expected level.

Process Development and Physical Property Characterization

Progress has been made in analyzing selected samples from the ANG test system toward better understanding its performance. Three monoliths were selected from the assembly, one from an end of the assembly, another from about one-fourth of the way through the assembly, and one from the center. Each monolith was sectioned and machined, providing test specimens for the Oak Ridge National Laboratory (ORNL) methane adsorption test apparatus (MATA), shown in Figure 1. Data were accumulated on the first specimen, indicating a best case V/V (liters of gas per liter of tank volume) of 96-97 rather than the 140-150 V/V range projected for the system. Results indicated uniform density of material from the inner regions outward to the edges, resolving questions about possible variations in density such that only portions of the monolith volume were active in methane adsorption. Because each monolith was made using the same formula and with the same process, and each was activated similarly (although with some variations in final activation), differences in density of material producing inactive adsorption zones could account for lower-than-expected performance. Densitometry results confirmed uniformity of monolith density.

A new carbon fiber material with different properties was acquired. The material previously used contained fibers of solid cross section. SEM and TEM examination revealed that only a thin, outer layer of fiber was activated; the remainder of the fiber, about 80% or more of cross section, was not activated and therefore inactive in adsorption (Figure 2). The new fiber material included hollow fibers. Although they were of poor uniformity and a mixture of components compared with the refined material used in monolith formulation and tested in the ANG test system, use of the hollow fibers introduces the potential of increased surface area for activation. Development and testing of specimens with the new material is currently under way. A second type of new, high-quality carbon fiber material has been ordered; preactivated fibers are projected to increase the monolith surface area, thus increasing total adsorption.

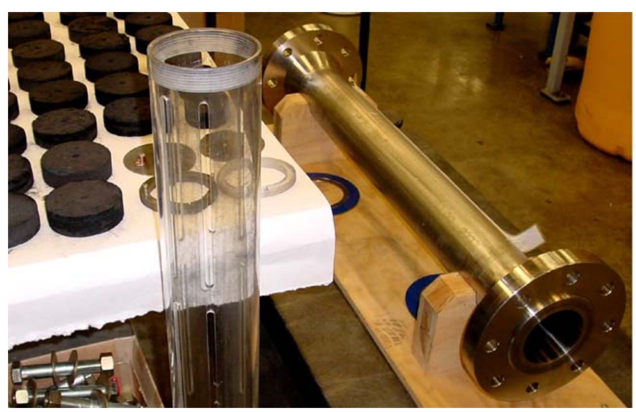


Figure 1. The major parts of the MATA are the test specimens, the stainless steel cell, and the Plexiglas liner. The dimension of each test specimen (the "puck") is $\Phi 4.45$ *in.* $\times 1.21$ *in.*

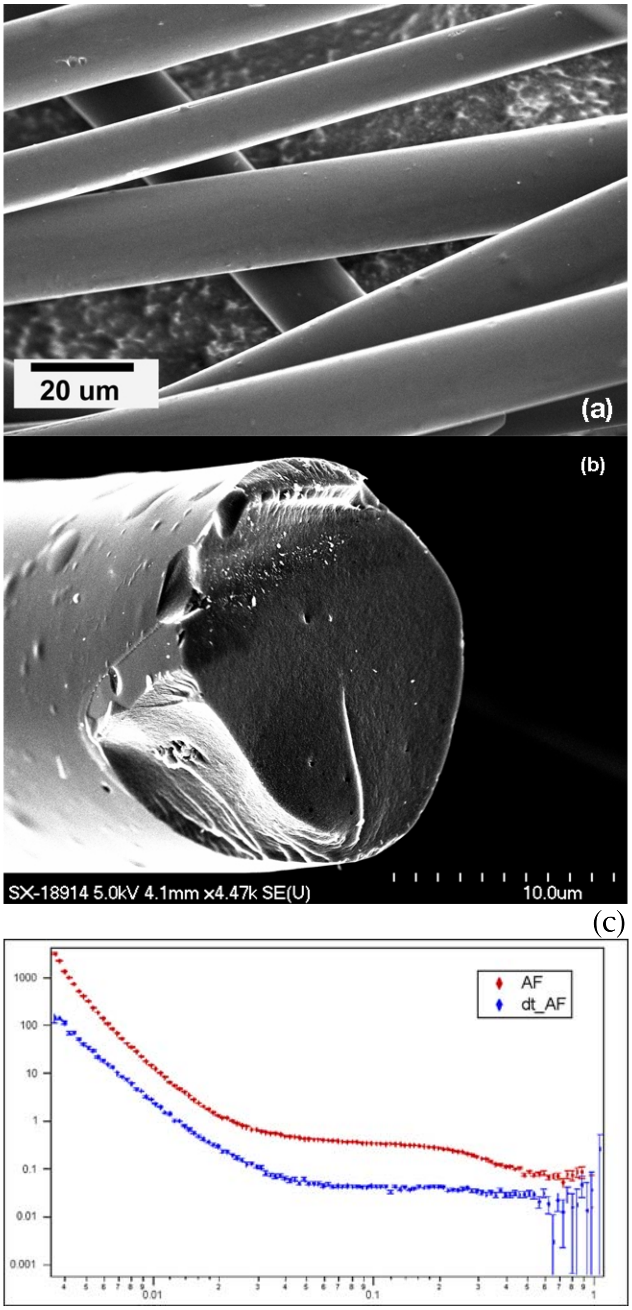


Figure 2. SEM images of the pre-activated Carboflex® fiber are shown in (a) and (b) SANS data shown in (c) of the Carboflex®. Deuterated sample is labeled "dt "

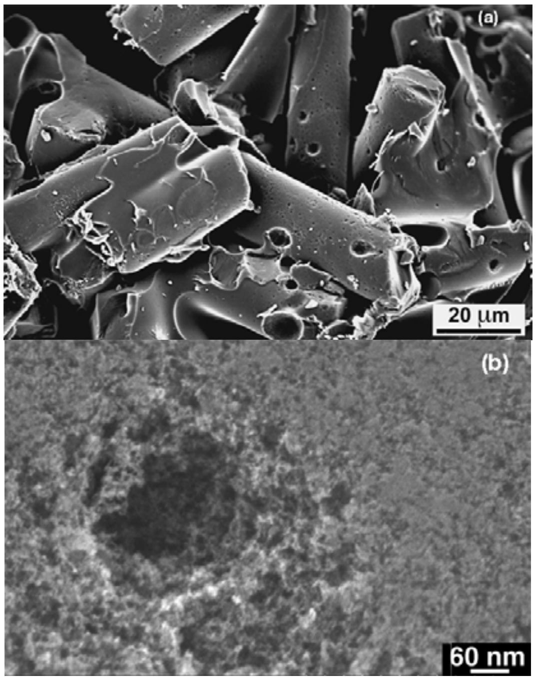
Materials Characterization

Characterization of carbon materials has been carried out using high-resolution TEM, SEM, SANS, and INS. The SANS study was undertaken at the Intense Pulsed Neutron Source at Argonne National Laboratory in early March 2003. The INS experiments were performed at High-Flux Isotope Reactor (HFIR) at ORNL

Experiments on Gas Storage Carbon

Carbon monoliths SMS48, SMM19, and Carboflex®, an activated pitch-derived carbon fiber, are the materials of interest. The contrast matching agent was deuterated toluene (chemical formula: C_6D_5-CD_3). The objective of the contrast matching SANS was to evaluate the open and closed microporosity of the gas storage carbon materials.

Figure 2 contains two SEM images of the activated Carboflex fibers. Images of SMS44 are included in Figure 3 because its microstructure is very similar to that of SMM19 and SMS48. The microstructure of Carboflex is drastically different from that of the carbon monoliths. Figures 2 and 3 also contain the SANS data for Carboflex, and SMS48 plotted in log-log scale. Again, the structural difference is revealed in the SANS scattering curves. As expected, each deuterated sample has much lower scattering intensity at the high-q region, suggesting that the pores have been filled with toluene. Results of the SANS data analysis are summarized in the following discussion. The average micropore size of the Carboflex is 1.5 nm, slightly larger than the two monoliths at 1.3 nm. The microporosity (for all the pores of less than 2 nm) is 9% and 10% for the SMM19 and SMS48, respectively. The microporosity of the Carboflex is 6%. The deuterated samples have very low closed porosity (<0.2%). Considering the margin of error, it is concluded that these materials have no closed micropores. In addition, because toluene penetrates into the micropores, all three deuterated samples show an increase of average pore size to 2.9 nm. Estimated error is about 15% for all the reported data.



(c)

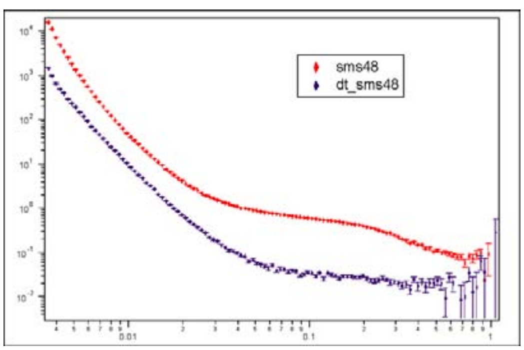


Figure 3. SEM images of the monolith SMS44 are shown in (a) and (b). Its structure is very similar to that of SMM19 and SMS48. SANS data for the carbon monolith SMS44 are shown in(c).monolith SMS44 are shown in(c).

Monolith in Methane

INS of the gas storage carbon monolith (NGS003) was carried out on the triple-axis spectrometer, HB1 at HFIR in October 2002. It was a proof-of-principle experiment that demonstrated that INS experiments can be performed on storage material. The study showed that no preferred adsorption sites exist on the carbon surface. A more detailed study on a modified setup will be undertaken in FY 2004.

Modeling and Simulation

Computer simulation work has been initiated using a reverse Monte Carlo (RMC) approach. It is imperative to conduct the calculation on a structure model that resembles the true features of the carbon material. Based upon this project's high-resolution TEM work, such a model is constructed with sufficiently large size and is filled with spheroidal interconnecting micropores. The initial calculation indicates that the pair distribution function (PDF) of the model matches reasonably well with the experimental data found in the literature. Figure 4 contains a sketch of the models and their radial distribution functions.

Prototype Tank Design Construction and Testing

Part 1

A series of 12 experiments were performed on samples machined from carbon monoliths selected from the Big Tank (Figure 5), along with additional tests using a modified experimental procedure (see discussion in Part 2). The methodology for performing each test used the existing guideline, MET-CIMT-SOG-124.

Adsorption performance indicates repeatability with an average of 3.58 g (+0.185g; -0.143 g) at 500 psi for an average of 8.77 wt %, and 4.413 g (+0.595 g; -0.138 g) at 900 psi for an average 9.78 wt %. Increasing fill pressure to 900 psi, +80%, increases stored methane by just 23%, which was accounted for mostly as adiabatic compression rather than adsorption. Accounting for methane retained in the carbon, deliverable methane averaged 88.07 V/V at 500 psi and 117.36 V/V at 900 psi. Retained methane averaged about 15% for all tests at 500 psi and about 20% for the 900 psi tests, indicating that subsequent deliverable

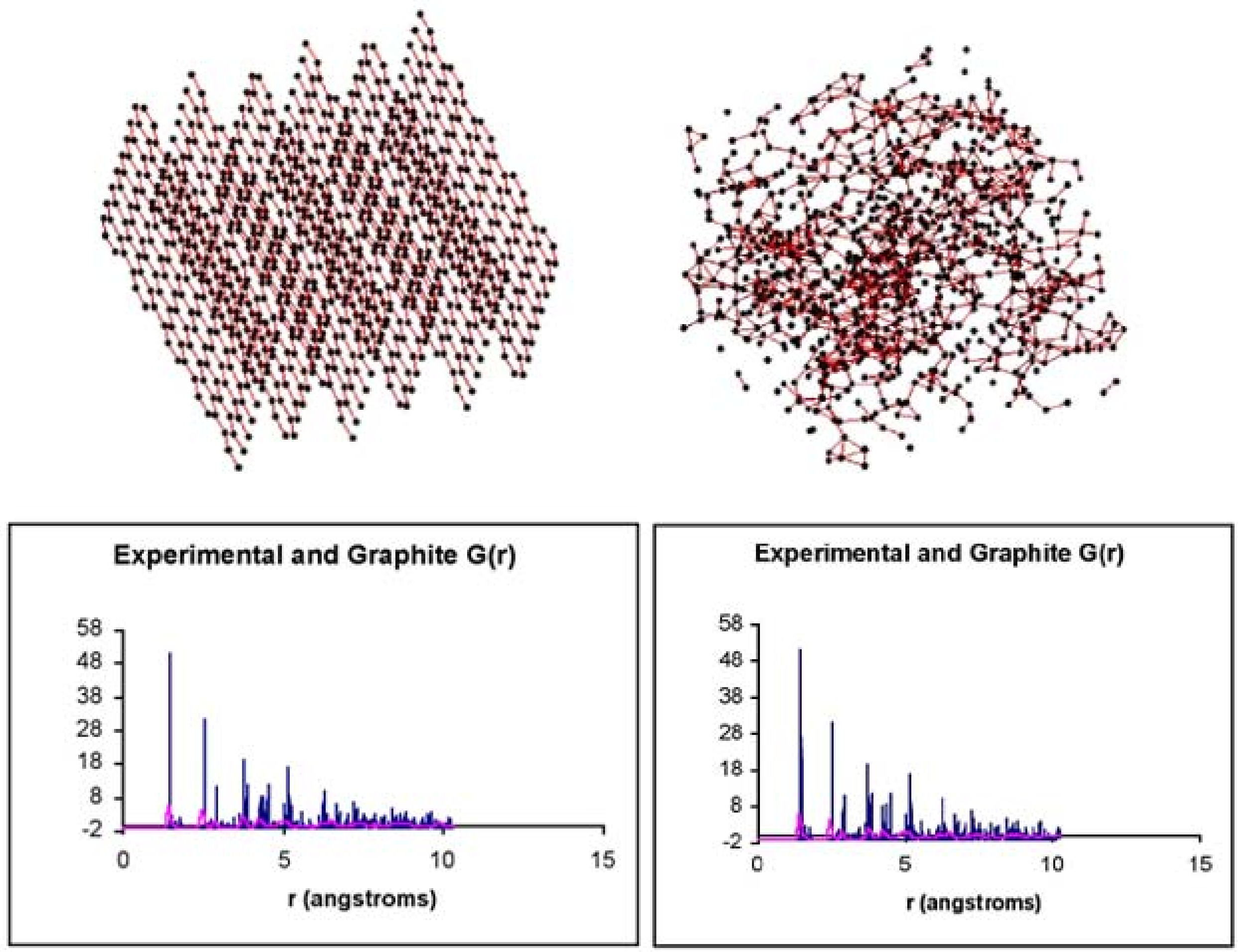


Figure 4. Graphite or disordered carbon is the initial starting structure for RMC simulation (experimental curves are the lighter, smaller peaks in the chart): (a) the graphite structure and its radial distribution function (RDF)and (b) disordered carbon and its RDF. The experimental peaks broadened and shifted relative to graphite.

charges of methane under ambient conditions, without prior vacuum extraction of residual gas in the carbon, is reduced proportionately. The potential of second and subsequent charges is expected to deliver about 75 V/V at 500 psi and 94 V/V at 900 psi.

When methane is introduced to the specimen, adsorption is rapid, as indicated by temperature peaking within 2–3 min. Rapid adsorption of methane is also supported by no additional methane take-up during specimen exposure to elevated pressures over times up to 1.5 h. Rapid saturation of specimens indicates that increasing specimen density may produce improved storage capacity. If adsorption is linear with density, increasing the density by 30%, from the tested average of 0.61 g/cc to a proposed density of 0.8 g/cc, will likely increase adsorbed methane at 500 psi to the range of 900 psi performance, ~4.65 g or ~10 wt %, from the same BET material and the same burn-off, while having little affect on adsorption time. Such a linear relationship suggests that 0.95 g/cc may deliver 135 V/V at 500 psi and 181 V/V at 900 psi; the later achieves the currently targeted DOE milestone. With the new pre-activated fibers received recently, along with the prospects of additional activation after specimens are made, higher-performance monoliths may result. A second set of monoliths constructed of hollow fibers, or fibers with increased external surface area, is likely to add to performance.

The research team recommends performing a similar series of experiments using the new materials and the established monolith fabrication method used for making the Big Tank material. In addition, the research team recommends that a second set of specimens be made from sample material with some hollow fibers, or fibers of increased external surface area. These fibers may further enhance performance.

Part 2

A series of pressure tests were conducted on one specimen with the density of 0.645 g/cc to examine the effects of exposing "clean" specimens (heated and evacuated as prescribed by the guideline) to pressurized methane in charges of 0–100, 0–200, 0–300 psi, etc., to reveal the characteristics of heat of adsorption and incremental adsorption. This experiment indicates that the performance is predictable from isotherm plots for this material, and that beyond about 200 psi, additional loading as adsorbed methane is slight. The 0–200 psi produces the greatest heating effects and accounts for 73% of contained methane at 500 psi. Isotherm plots show rather linear adsorption after rapid initial loading, indicating that available sites for adsorption are largely filled when they are initially exposed to methane. Retained methane of ~19% (in the 500 psi case) indicates that with discharge, many adsorption sites retain methane such that second and subsequent chargings yield proportionately reduced deliverable methane. Results from this method, 89.6 V/V at 500 psi, agree with deliverable methane data from the previous tests, M_D = 88.07 V/V, within 2%.

As noted in the above recommendation, the second method also indicates that increasing density may produce greater adsorption and greater deliverable yield. If adsorption is linear with density, increasing specimen density to 0.95 g/cc translates to the deliverable volume at 500 psi of M_D = 132 V/V. If activation is also linear, increasing activation from 1300 m^2/g to about 1500 m^2/g may yield stored volumes of 152 V/V, further supporting the recommendation to fabricate and test new monoliths made from newly acquired material. To achieve the DOE milestone of 180 V/V with specimens of 0.95 m^2/g, assuming linear relationships, activation to about 1775 m^2/g is required.

The research team recommends that monoliths be made from new material and activation be attempted at near 1800 m^2/g. Then testing should be repeated as outlined.

Publications

Tim Burchell, Jane Howe, Alex Gabbard, and Mike Rogers, "Adsorbent Carbon Fiber Composites for the Storage of Natural Gas," SAMPE 2003. (Third place winner and winner of the award Best Paper of SAMPE 2003.)

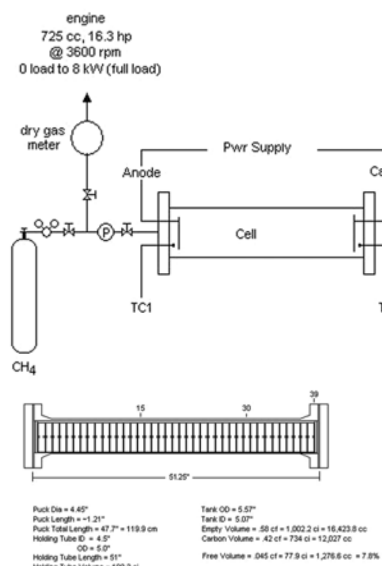


Figure 5. (a) Schematics of the ORNL MATA and (b) the dimension of the gas cell, the Big Tank.